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NON-OZONE DEPLETING SUPERCRITICAL CLEANING FLUIDS: DESIGN, FABRICATION, AND OPERATION OF FIRST PREPRODUCTION NATURAL CONVECTION DEVICE

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Southwest Research Institute
6220 Culebra Road, P.O. Drawer 28510
San Antonio, Texas 78228
Telephone: 210-684-5111 Fax: 210-522-3649

MATERIALS DIRECTORATE
WRIGHT LABORATORY
AIR FORCE MATERIEL COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OH 45433-7734

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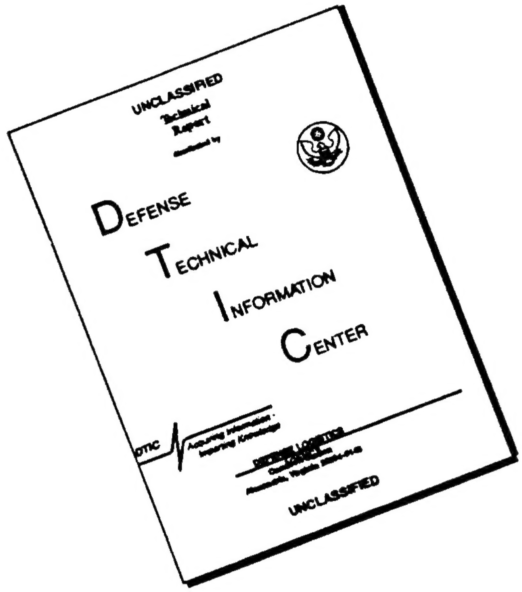

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Mark D. Kistner
MARK D. KISTNER
MATERIALS BEHAVIOR & EVAL SECTION
MATERIALS ENGINEERING BRANCH
SYSTEMS SUPPORT DIVISION

James L. Folck
JAMES L. FOLCK, TAM
MATERIALS BEHAVIOR & EVAL SECT
MATERIALS ENGINEERING BRANCH
SYSTEMS SUPPORT DIVISION

Theodore J. Reinhart
THEODORE J. REINHART, CHIEF
MATERIALS ENGINEERING BRANCH
SYSTEMS SUPPORT DIVISION

Gary K. Waggoner
GARY K. WAGGONER
CHIEF
SYSTEMS SUPPORT DIVISION

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Mary Marshall , W. Thomas Roberds
Gordon Pollard

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6220 Culebra Road, P.O. Drawer 28510
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ABSTRACT

In accordance with the 1987 Montreal Protocol and the Clean Air Act Amendments of 1990, a production ban on Class 1 ozone-depleting substances becomes effective 31 December 1995 in the United States. The production ban includes two cleaning solvents (1,1,1-trichloroethane and Freon 113) which are mission critical to numerous USAF weapon systems. The DoD stockpile contains 350,000 lbs. of 1,1,1-trichloroethane and 500,000 pounds of Freon 113 to last through the year 2000, but alternative cleaning solutions are required since the stockpile will eventually be depleted.

Southwest Research Institute (SwRI) has developed the natural convection supercritical fluids (SCF) cleaner as a potential substitute for 1,1,1-trichloroethane and Freon 113. This cleaner offers the potential for reduced capital investment, lower operating costs, and improved maintainability and reliability over the conventional SCF devices principally by eliminating the circulating SCF and the compressor requirement. The natural convection system, however, can deposit "drag out" solute onto the part during decompression.

The first prototype cleaner (only 2 inches in diameter) was built in 1991 at SwRI on internal research and development funds. To demonstrate the natural convection technology on a production scale and to provide the USAF with a useful tool for detailed assessments, this project's objective was to construct a preproduction device which could clean 70% of all parts handled at WPAFB (Wright-Patterson Air Force Base) Aircraft Modification Directorate in Building 5. Thus, a 10-inch diameter cleaning chamber was specified to handle parts weighing up to 70 pounds. The SwRI prototype was used for experimental studies for process design purposes and as a guide for scale-up.

The preproduction device has been designed, fabricated, assembled, tested, debugged, and operated. Complete drawing packages, design calculations for the pressure vessel, facility requirements, and instrumentation schematics have been submitted to the USAF. Several parts have been cleaned and computational fluid dynamics model validation studies have been conducted. The system is ready for technology transfer, and several equipment manufacturers have approached SwRI for commercialization discussions. The device will remain for several more months at SwRI for field testing with parts received from the aerospace community to assess performance for several specific applications.

This report documents the design decisions, provides the lessons learned, and reviews the preproduction device components and specifications. Guidance for future research and trade-off decisions for subsequent natural convection SCF cleaners is also presented.

EXECUTIVE SUMMARY

Southwest Research Institute (SwRI) was contracted from 15 November 1993 through 15 December 1995 by the University of Dayton Research Institute (Subcontract No. RI-22377X) to design and construct a "non-ozone depleting supercritical cleaning fluids" chamber in San Antonio, Texas. The end client (Prime) for this system is Wright-Patterson Air Force Base (WPAFB), Ohio. This program is being conducted under their Contract No. F33615-89-C-5643.

The supercritical cleaning fluids chamber is a pressure vessel used to degrease parts. It operates at pressures up to 2,000 psig and temperatures up to 200 °F, and supercritical carbon dioxide is the cleaning solvent eliminating the need for ozone-depleting substances. The unit will clean parts as large as 8.25 inches in diameter and 12 inches long (representing 70% of all parts cleaned at WPAFB Aircraft Modification Directorate, formally the DMMF facility).

The cleaning mechanism (U.S. Patent 5,401,322 issued to SwRI on 3/28/95) is attributed to equilibrium solubility curves; in supercritical fluids, contaminant solubility generally decreases with increasing temperature near the critical pressure. The contaminated part is placed in the low temperature zone, where the solubility of contaminant is high. After the contaminant has dissolved, the contaminant-rich solvent flows by gravity to the high temperature zone, where the solubility of contaminant is low. The solvent is replenished in the hot zone by the reduced contaminant solubility which causes contaminant phase separation.

A multidisciplinary team was used to design, construct, and start-up the preproduction cleaner. Specialty areas included pressure vessel technology, user-friendly computer control, and computational fluid dynamics modeling. The SwRI 2-inch diameter prototype cleaner was used as the design basis and for experimental trials to gather design data. To minimize cost and risk, many commercially available components were included in the preproduction cleaner. Field modifications (mechanical, electrical, and software upgrades) were made during both assembly and operation to repair unreliable subsystems or to make the cleaner easier to operate.

The technology is versatile, and prospects for technology transfer to manufacturers of commercial degreasers are promising. A dual-use opportunity has also arisen to apply this technology to dry cleaning. SwRI is pursuing these patent licensing opportunities.

The cleaner is being operated under another subcontract to gather additional cleaning data for a range of parts and contaminants received from the Air Force and the aerospace industry.

The scale-up of this new technology was successful under the UDRI subcontract. The completed preproduction cleaner is fully debugged and tested. It is user-friendly, easy to operate, safe, quiet, and non-polluting.

CREDITS AND ACKNOWLEDGMENTS

The University of Dayton Research Institute provided the subcontract for this research. We appreciate the efforts and patience of **Mr. Robert Askins**, Program Manager, and **Ms. Carol Eckley**, Subcontract Specialist, in managing this subcontract.

Many technical people from Wright-Patterson Air Force Base (WPAFB) were responsible for the success of this project. **Mr. Ted Reinhart** provided the initial vision for the natural convection process based upon some crude blueprints of a prototype 2-inch diameter vessel. Mr. Reinhart's continual excitement, technical oversight, and internal proactive approach for funding has allowed the project to move at a good developmental pace without contractual disruptions. His direction has provided the necessary constancy of purpose to maintain the original work scope while permitting several technical excursions (such as computational fluid dynamics modeling funded under another subcontract) which were essential to solve unforeseen technical challenges.

Mr. Charles Pellerin assisted the technology transfer efforts through discussions with his numerous business associates to locate a visible, accessible, and viable facility for long-term operation and assessment of the preproduction device. **Mr. Phil Mykytiuk** has kept up with the project details to ensure that the cleaner specifications can accomplish the Air Force goals. His enthusiasm for the new cleaner and knowledge of the cleanliness issues has kept the project well focused.

Dr. Harvey Paige was exceptionally generous with his time and knowledge of supercritical fluids. He volunteered to assist with this project since he has the most chemistry knowledge in the field at Wright Laboratories. Dr. Paige was an invaluable mentor offering practical improvements and recommending important technical studies, the most significant of which was the computational fluid dynamics modeling and experimental validation work.

Mr. Mayank Patel was a constant force in visioning the commercialization of this device. He single-handedly commandeered (procured funding and instilled enthusiasm) the first design review meeting in San Antonio. This meeting was instrumental in moving the project into a good deliverable item for the USAF since it provided the necessary user interface requirements to build it to the customer needs (i.e., the Aircraft Modification Directorate at WPAFB whose facility performs numerous cleaning operations and whose staff understands the substitution cleaner operational specifications).

Messrs. **Jack Flute** and **Ron Case** were great advocates for this research program and provided the necessary questions which resulted in significant project cost savings and improved operational performance by the major design change from 2 to 1 large ball valve ($70,000 unit cost).

Messrs. Mark Forte, Chuck Deluise, Bob Urzi, and **Mark Kistner** were USAF program managers at early stages of the design and development efforts. Their patience and advise during these less directed phases allowed the innovative solutions to build a viable preproduction device.

Southwest Research Institute (SwRI) coordinated a team of experts from numerous operating segments of the organization. Contributions came from the Departments of Applied Chemistry and Chemical Engineering, Automation Engineering, Environmental Engineering, Electronic Systems, Marine Technology (pressure vessel design, fabrication and testing technology), Fluids Engineering, and Institute Quality Assurance. The SwRI team included highly trained and experienced scientists, engineers,

designers, technicians, welders, and quality assurance specialists. Each individual made key contributions necessary for the success of this project. Their activities described in the following paragraphs can never represent the dedication and pride of good work demonstrated by each person.

As the inventor of the natural convection device, I'm forever indebted to their endeavors, for without these people, the device would never have been built. I will always remember the joy of working in this team and the tremendous commitment towards making this system function well with high quality, excellent safety, and good reliability.

Mr. Gordon Pollard, Designer, conducted the pressure vessel detailed design, directed the procurement and installation of all mechanical systems (valves, closures, fittings, hubs, piping, etc), coordinated the fabrication work with drawing compliance and resolved resulting conflicts, led the assembly and field modifications, and did the rework and reliability and maintainability improvements on the basket transfer system. Mr. Pollard's superb document control system, high quality and attention to detail, tremendous time management skills and quick response to difficulties, and tremendous ability to work within a team has been the key to the success of this preproduction device. He acted as project manager on numerous occasions to keep the schedule moving, especially during fabrication when the project manager was out of town. He also identified many potential "show-stoppers" and brought these with possible solutions to the attention of project personnel with plenty of time to correct the situation.

Mr. W. Tom Roberds, III, Senior Research Engineer, Department of Electronic Systems, developed the control logic for the preproduction unit and all of the automated sequences, identified and programmed the safety interlocks and software checks to prevent the operator from creating unsafe conditions, and designed and installed the electrical and instrumentation wiring. The operating manual, start-up and operational check-out, training, debugging, and what-if hazard assessments were all led by Mr. Roberds. Mr. Roberds has a natural ability to extract the best out of everyone he works with, has the patience to troubleshoot problems and get the responsible vendor to repair their equipment, and is extremely competent with computer programming, electrical wiring schematics design, and field work. Mr. Roberds met every deadline, immediately responded to field needs for changes to the computer code or electrical additions and has been invaluable during the system debugging tasks. Success on this project is attributable to Mr. Roberds and Mr. Pollard.

Mr. David Morales, Technician, was our lead operator and was intimately involved with field assembly, operational check-out and demonstrations, experimental data gathering, and troubleshooting this system. His responsiveness, good technical understanding, foraging capability, and flexibility especially to long evening work hours have been a great asset to this project.

Mr. Lawrence Goland, P.E., Manager, ensured that the pressure vessel adhered to ASME code by directing the design, making the engineering computations and providing drawing approvals. He was an able and candid advisor on the operability of system and quality of the final delivered product.

Mr. Ben Delgado, Sr. Technician, installed the instrumentation wiring and assembled the controls enclosures. Ben's attention to detail and careful installation permitted a smooth startup since the lines were properly labeled and connectivity was verified before the wires were put into conduits.

Mr. Charles Smith, Quality Assurance Technologist, and **Bob Young,** Senior Quality Insurance Inspector, conducted the Quality Assurance inspections of incoming materials for American Society of

Mechanical Engineers (ASME) code compliance, observed fabrication for conformance with the production drawings, coordinated and reviewed the dye penetrant and x-ray inspections of each weld, wrote the non-compliance reports, and resolved issues requiring corrective action. The exactness of their work, sense of urgency for inspections for minimal schedule impact, and their gracious approach created the teamwork necessary to produce excellent pressure vessel components.

Mr. Louis Kim, Research Engineer, developed the piping and instrumentation diagrams (P&ID), specified the liquid syphon system, and designed the internal heaters. **Mr. A. C. Rogers** specified the thermoelectric cooler for elimination of all ozone-depleting refrigerants. Mr. Rogers found **Dr. Yong Nak Lee**, President, Heat Transfer Research and Development, Ltd. who specially designed the highly efficient thermoelectric cooler for us. **Dr. Niraj Vasishtha**, Research Engineer, assisted with polymer compatibility specifications for seals and O-rings as well as calculating energy balances for various fluid dynamics scenarios.

Mr. Glynn Bartlett, Automatic Mechanical Systems, developed the concept and first version of basket transfer system. He worked with the SwRI model shop and machine shop to resolve component fit issues. His manager, **Mr. James Luckemeyer**, was an excellent facilitator to ensure that details were resolved early and cost was monitored regularly.

Messrs. Frank Garcia and Larry Bybee were the welders on the pressure vessel. **Mr. Francis Caroline**, Shop Foreman, and **Mr. Doyle Smith**, Fabrication Supervisor, oversaw the fabrication of the pressure vessel to conform to code compliance and adherence to production drawings. Dedication, professionalism, and commitment to low cost with high quality was exemplified in all of these men.

The Division 15 Model Shop and SwRI Machine Shop produced high quality items used in various subsystems of the SCF (supercritical fluid) cleaner. Their quick response and ability to meet our sometimes very tight deadlines is sincerely appreciated.

The SwRI Pressure Laboratory has graciously provided a large pit, complete with reinforced three-foot thick concrete walls, for the prove-out and operation of the preproduction device. **Mr. Jesse Ramon**, Manager, provided excellent support, and his staff members were always of tremendous service. Special recognition is given to **Messrs. Joe Canellis**, Senior Technician, **Robert West**, Senior Technician, and **Mark Mercer**, Senior Technician, for assembly (especially the task of torquing the Grayloc bolts to 870 ft.-lbs.) and constant safety reviews.

Mr. James R. Scott, Senior Research Engineer, CIH, was regularly consulted on employee health and safety issues. Jim was the sole colleague to discuss the initial invention concepts in the late 1980s and always provided practical and insightful advice. **Mr. Jac Harding**, Safety Officer, provided operational safety reviews and corrective action decision-making advice during operational mishaps. Secretarial support was ably provided by **Mss. Jackye Moravits, Brenda Honeyager, Jackie Bean,** and **Debbie Podnar**. Their long hours and project dedication is always appreciated.

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LIST OF ABBREVIATIONS

ANSI	American National Standards Institute
CFCs	Chlorinated Fluorocarbons
CFD	Computational Fluid Dynamics
EPA	Environmental Protection Agency
JAST	Joint Association for Supercritical Technologies
LAI	Lawrence Associates Inc.
MEK	Methyl Ethyl Ketone
ODS	Ozone-Depleting Substances
SC-CO_2	Supercritical Carbon Dioxide
SwRI	Southwest Research Institute
UDRI	University of Dayton Research Institute
VOC	Volatile Organic Compound
WPAFB	Wright-Patterson Air Force Base

I. INTRODUCTION

The developmental effort described in this final report was needed because of new environmental laws which have forced the elimination of excellent cleaning solvents. An overview of these changes and the reasons for pursuing the technology of supercritical cleaning fluids are given in the following pages.

A. The Montreal Protocol Demands Substitutes for Ozone-Depleting Substances (ODS)

Many chlorofluorocarbons (CFCs) destroy the ozone layer of the earth's stratosphere. The Montreal Protocol calls for a worldwide effort to phase out the use of all fully-halogenated CFCs in an effort to preserve our environment. The original Montreal Protocol was signed in 1987 and ratified by 64 countries. The Clean Air Act Amendments of 1990 mandate a production ban on Class 1 ODS materials effective 31 December 1995 in the United States.

ODS materials being banned from production have numerous applications in the USAF including refrigerants, fire extinguishers, solvent cleaners, paint solvents, and adhesives. The USAF, EPA, DOE, and production shops in both small and large businesses have been substituting non-ODS materials for these applications. Substitution successes have been numerous as evidenced by the reduction in 1,1,1-trichloroethane purchases in 1995; the USAF purchased only 14% of 1,1,1-trichloroethane purchased in 1992.[1]

The DoD recognized several years ago that specialized requirements for weapon system readiness might not have acceptable technology to eliminate the need for ODS materials, so two approaches were taken: (1) invest funds into research for new technologies, and (2) stockpile ODSs to establish a reserve for "mission-critical" requirements through and beyond the year 2000, allowing time for research and development advances.

The USAF estimates that 19% of the ODS usage for which adequate substitutes are unavailable is for cleaning. Of the cleaning applications, 35% is for metals, 40% for electronics, 12% for oxygen systems, and 13% for standards. In addition, five of the top fifteen solvent-related USAF needs (see Table 1) directly deal with cleaning.[2] To maintain mission readiness, the DoD Reserve will contain 350,000 pounds of 1,1,1-trichloroethane (also known as methyl chloroform) for USAF cleaning requirements and 500,000 pounds of CFC-113 for USAF and Navy cleaning needs.[3] Consequently, the need for new technology is crucial to the USAF.

B. Supercritical Carbon Dioxide as a Replacement Solvent

Much work has been completed to demonstrate the benefits of SC-CO_2 cleaning. The advantages are based on environmental aspects, excellent solubility for organic compounds, and low operating costs.

TABLE 1. TOP ODS SUBSTITUTION NEEDS FOR USAF CLEANING APPLICATIONS

Need No.	Description	Rank in Top 15 Solvent-Related Needs
129	Heavy degreaser on aluminum surfaces, carbon removal, and a pre-paint and pre-adhesive cleaner.	2
991	Non-ODS/Non-EPA 17 drop-in replacement for 1,1,1-trichloroethane used in oxygen-tube cleaning	5
103	Replacement for 1,1,1-trichloroethane and MEK as wipe solvents and inclusion into Tech Data and MIL-SPECs.	6
429	Replacement cleaner/drying agent for applications where parts could be exposed to nitrogen tetroxide (oxidizer).	10
203	Eliminate or reduce VOC emissions and/or hazardous waste generation from solvents used to clean metal parts.	11

1. SC-CO_2 is Environmentally Friendly

SC-CO_2 as a cleaning solvent has both environmental and performance improvements over chlorinated fluorocarbons (CFCs). It is a nontoxic and nonflammable solvent, has no solvent waste, and has no reportable emissions. SC-CO_2 is not a CFC, it is not an ODS, and it is not on EPA's list of 17 most toxic chemicals. SCF can work better than other solvents including aqueous cleaning systems since there is no solvent residue, no parts drying, and no dilution of the contaminant waste. The operating cost of SC-CO_2 is low since the solvent cost is only $0.06/lb-$0.15/lb (depending on bulk size purchased).

2. SC-CO_2 has Good Cleaning Performance

Much has been published on the efficiency of SC-CO_2 for cleaning various components. For example, Pacific Northwest Laboratory has successfully cleaned a variety of metal parts and electromechanical systems.[4] Phasex Corporation has removed contaminants including hydrocarbons, oils, esters, and silicones from parts ranging from gyroscopes to porous ceramics to laser optics parts.[5]

SC-CO_2 can perform better than CFCs for ultra-small pores and interstices since the SCF has very low solvent surface tensions. Also, operating temperatures for SCF are moderate (30-80 °C) and tend not to damage many components.

3. Few SCF Commercial Cleaners Exist

SCF cleaning technology is not the eureka since it does have disadvantages which include high capital costs associated with the stainless steel pressure vessel (operating pressures from 1,000 to

6,000 psi), corrosion in the presence of water (requiring stainless steel construction), and parts damage from the pressure cycles (either crushing sealed chambers or de-laminating coatings). Consequently, the cleaning industry typically recommends SCF as a last resort after proving that aqueous cleaners and no-clean options aren't feasible. Thus, there is a limited market for the SCF cleaners.

There are many specialty applications ranging from gyroscopes to electronics which the USAF faces in which aqueous cleaners and no-clean options won't work. Numerous SCF demonstration efforts exist using the flow-through process which have successfully cleaned parts but which also depend on expensive compressors to maintain the SC-CO_2 state. Nonetheless, several commercial flow-through SC-CO_2 units have been sold by Hughes Aircraft and DeFlex companies demonstrating a market need.

C. Natural Convection SC-CO_2 Cleaner

Southwest Research Institute (SwRI) invented a new process for using SC-CO_2 in a cleaner without continuously compressing carbon dioxide above the critical conditions (1,070 psi and 31 °C) and running it past the dirty part until all of the contaminant was solubilized. The SwRI alternative technology is schematically presented in Figure 1 and is called the SC-CO_2 natural convection cleaner.

Liquid carbon dioxide (CO_2) is syphoned into the cleaner by creating a slight pressure drop via the CO_2 vent valve. Once sufficient liquid CO_2 is added to the vessel, electrical resistance heaters raise the temperature and, consequently, the pressure into the supercritical fluid zone. Thus, the expensive compressors and support equipment necessary in flow-through SC-CO_2 cleaners are eliminated. In addition, the power requirements are less for the natural convection cleaner and no hearing protection is needed since noise levels during normal operation are low.

D. USAF Funded Development of Natural Convection Cleaner

SwRI built a prototype device for 1½- inch parts under internal research funding. Based upon knowledge gained from the prototype, the USAF (specifically, the Materials Directorate of Wright Laboratories) funded SwRI to design, build, and operate a preproduction natural convection cleaner through two subcontracts. The University of Dayton Research Institute (UDRI) subcontract was to design and construct a cleaner for items which will fit into a 10-inch diameter and 11-inch long cleaning area. The Lawrence Associates Inc. (LAI) subcontract was to study the fluid dynamics flow patterns within the cleaner, operate the cleaner to understand the range of contaminants and cleanliness levels applicable, develop operating manuals, conduct training of USAF personnel, and to develop a sampling system to automatically determine when a part is clean independent of the contaminant(s), quantity of dirt, or shape of the part. The USAF goal was to obtain a new cleaner usable for 70% of the parts at the DMMF facility at Wright-Patterson Air Force Base (WPAFB) which could demonstrate the utility of the natural convection SC-CO_2 technology. This report describes the accomplishments of the UDRI subcontract.

II. OBJECTIVE

To demonstrate the natural convection technology and to provide the USAF with a useful tool for detailed assessments, this project's objective was to construct a preproduction device which could clean 70% of all parts handled at the WPAFB Aircraft Modification Directorate in Building 5 (formerly known as DMMF). Thus, a 10-inch minimum diameter cleaning chamber was specified for parts as large as 8.25 inches in diameter and 12 inches long with weights up to 70 pounds. The first prototype cleaner (only 2 inches in diameter) existed at SwRI and had been built on SwRI internal research and development funds. This SwRI prototype was the basis of the scale-up and was used for experimental studies to assist process design.

Since carbon dioxide becomes supercritical at 1070 psi and 31 °C, the cleaner had to conform to pressure vessel codes. The vessel was designed for a lifetime of approximately five years of continuous usage in the production shops. (Pressure vessel lifetimes are based upon the degree of fatigue undergone by the metal through pressurization and depressurization cycles; the computer system maintains the usage log so that the remaining life can be readily calculated.)

The preproduction unit was designed for operation at an USAF facility using regular equipment operators, unskilled in the chemistry of supercritical fluids. Consequently, the reliability of the device and ease of use were imperative. A single computer is used to collect all data and to control the cleaning operation. Automatic sequences have been established so the operator selects only the length of time for cleaning and the degree of rotation (none, continuous, or 90° turns) of the parts basket, loads the parts basket, then selects "start clean." When prompted by the automated cycle, the operator starts and also terminates filling the transfer chamber with liquid CO_2. Cleaning then automatically proceeds without further operator intervention. When the operator returns, the parts are ready for removal from the basket.

The long lifetime of the vessel (approximately 30,000 basket loads of parts) will permit cleaning of a large variety of parts (numerous shapes, configurations, materials, and contaminants) to determine its versatility and limitations. SwRI will develop operating guidelines (recommended temperatures, pressures, and dwell times) based on the experimental programs funded under the LAI subcontract. The preproduction cleaner will eventually be placed in a production shop for everyday cleaning needs; the robustness and operational ease will be determined by the shop workers.

III. CONCLUSIONS

Under this UDRI subcontract, the natural convection SC-CO_2 cleaner was demonstrated. The effort showed that fabrication of a fully-automated preproduction device was possible. Many commercially-available components were used in the fabrication for easy technology transfer in assembling future similar units.

SwRI used many technical disciplines ranging from mechanical designers to chemical and electrical engineers in the design of this first preproduction unit. This multidisciplinary design team foresaw potential integration problems and, thus, was able to correct deficiencies in the design stage with minimal technical and cost impact. In addition, the vast and complimentary experience of each design team member brought solutions forward which had been successfully implemented on other projects and which simplified the preproduction device. Finally, the reviews and comments received from USAF personnel (both at Wright Laboratories and at WPAFB Aircraft Modification Directorate) and the UDRI and LAI contractors aided the preproduction design by recognizing improvements or making small design changes to make the device readily acceptable by shop floor production workers.

A. Preproduction Device Works Per Specifications

The preproduction device is fully operational and has been demonstrated as simple to operate, safe, and quiet during normal operations. The entire system employs no ODS; even the chiller is a thermoelectric device to eliminate the use of refrigerants.

Several experiments have been conducted which prove that cleaning is occurring within the preproduction device's natural convection cleaning chamber. There are numerous operating variables for optimizing cleanliness; these include temperatures in the hot and cold zones, operating pressure, dwell time for the part, rotation of the part, natural convection flow pattern controlled by the baffle design, and co-solvents. More research is required to develop a guidance document for selecting values for these variables for numerous shapes, materials, and contaminant composition and quantity. The experimental work to date has also shown that oils do pool at the base of the cleaning column.

B. Cleaning Times and System Through-put Trade-offs Depend upon Application

The natural convection SCF cleaner has many options which permit an unlimited number of configurations. Future design decisions must evaluate each of these (cleaning cycle times, production through-put, cleaning chamber size, quantity of expendable CO_2, need for a transfer chamber and basket transport system) to optimize the device for its intended application. These decisions will affect the initial capital investment, operating cost, system complexity, and construction time and must be made for each application.

C. Process Patent Was Received

The science presented herein is novel as proven by U.S. Patent 5,401,322 issued March 28, 1995 and assigned to SwRI with royalty-free governmental use. Four technical presentations were given under the LAI subcontract, and the technical community gave general acceptance to this new technology. The preproduction system works for cleaning, and a range of contaminants, degree of cleanliness, and required

operating conditions for different-shaped parts and contaminants required by the technical community are being studied under the LAI subcontract.

D. Technology Holds Promise

Several promising technology transfer and dual-use opportunities have presented themselves as a consequence of building this preproduction device and demonstrating its operation. Several aerospace firms would like to evaluate the system to clean items like satellite heat pipes which currently require laborious efforts since Freon 113 was eliminated. Recently SwRI received a request to give an exclusive license to an SCF consultant to develop the system for dry-cleaning applications; negotiations are underway to define terms. Also, two major equipment manufacturers of degreasers have expressed interest in adding the natural convection SC-CO_2 technology to their existing lines of aqueous and solvent degreasers to provide an alternative technology. More experimental studies on the cleaning effectiveness are needed before serious discussions can begin with the equipment manufacturers.

This natural convection SC-CO_2 technology appears to be highly versatile with future equipment designs dependent on the application and the user's need for defined cleaning times, production throughput, types of contaminants, and various shapes and materials of parts.

IV. BACKGROUND

Many researchers have been studying the use of supercritical carbon dioxide ($SC\text{-}CO_2$) for a cleaning solvent to replace chlorinated fluorocarbons (CFCs) without relying on EPA 17 or other ozone-depleting substances (ODS) materials. Researchers include Rockwell International, Hughes Aircraft Company[6] and the "superScrub" device, the Joint Association for Supercritical Technologies (JAST) which includes Autoclave Engineers, CF Technologies, National Forge, Liquid Carbonic, and Los Alamos,[7] GCG Technologies Inc.,[8] and a cooperative European group consisting of Lund University and the Institute for Engineering Research in Goteborg.[9] All of these researchers have worked with a flow-through system for cleaning the parts.

SwRI has invented a new system[10] based upon natural convection and developed the first prototype under SwRI internal research and development program. The prototype vessel began operation in 1993 for parts less than 1.5 inches in any dimension (Figure 2 is a picture of the system). Under USAF funding from Wright Laboratory, Materials Directorate, a preproduction device has been constructed on this subcontract for parts as large as 8.25 inches in diameter and 12 inches long. Operation has successfully demonstrated cleaning without a compressor or supercritical fluid (SCF) pump.

A. Cleaning Capability

Much has been published on the cleaning efficiency of $SC\text{-}CO_2$ and the components which have been examined. Pacific Northwest Laboratory (operated by Battelle Memorial Institute for the U.S. DOE) has cleaned metal parts, machined parts, bearing assemblies, optical and laser components, precision mechanical parts, electromechanical systems, and medical devices[11]. Phasex Corporation has removed hydrocarbons, machining and lubricating oils, esters, silicones, Krytox, fluorocarbons, perfluoropolyethers, organosilicones, polychloro and bromotrifluoro-ethylenes, and halocarbon-substituted-triazines from parts including gyroscopes, accelerometers, porous ceramics and metals, thermal switches, electrical components, nuclear valve seals, laser optics parts, polymeric containers, electromechanical assemblers, and camera lenses. Phasex has also identified the highly compatible materials for $SC\text{-}CO_2$ to include stainless steel, beryllium, gold, silver, copper, ceramics, Teflon, silicone, and epoxy potting compounds.[12]

B. Flow-Through SCF Cleaners

Figure 3 depicts the main features of the flow-through SCF. The process includes a compressor, depressurization valves, the cleaning vessel, a waste separator vessel, an optical sensor for recycle control, and heaters to compensate for SCF expansion cooling. Cleaning efficiency has been improved with the use of internal stirrers to increase the turbulence inside the cleaning vessel and with ultrasonics.[13]

The cleaning times have been reported to be around 8 minutes,[14] but are a function of the contaminant characteristics and degree of cleanliness required. For example, Mr. Jackson reports a minimum of 45 minutes was needed to clean silicone insulated cables used in spacecraft electronic systems, as compared to 37 hours for conventional thermal vacuum bakeout processes.[15]

SCF flow rates have been reported to be range from 0.5 to 11 lb/hr.[16, 17] Operating conditions with pressures of 1500-6000 psi and temperatures of 50-80 °C have been reported.[18] High purity CO_2 enters the cleaning zone minimizing the opportunity for part contamination from the cleaning solvent so precision cleaning applications have been successful.

C. Natural Convection Cleaning

Supercritical fluid solvation power is modulated by varying the SCF temperature and pressure. Significant increases in solubility are achieved at the higher pressures, and consequently for flow through systems whose economics are directly related to the cleaning time, the greater the solubility, the less CO_2 needed and the shorter the time the compressor needs to operate.

To eliminate the flow-through SC-CO_2 requirements of the current cleaning technology, SwRI invented a means to continuously regenerate the SC-CO_2 within the pressure vessel. At pressures near the critical pressure (1070 psi for CO_2), the solubility of a compound decreases with increasing temperature (refer to Figure 4). Also, the density of the SC-CO_2 reduces with increasing temperature at a given pressure (refer to Figure 5). Thus, at isobaric conditions, the contaminant is solubilized in the cold region. The cold fluid with the contaminant then falls by gravity to a bottom heat exchanger at which point phase separation occurs (i.e., the contaminant separates from the hot, lower saturation concentration carbon dioxide). The hot SC-CO_2 rises since it is less dense, and the contaminant pools at the vessel floor since the contaminant (oils and greases) are more dense than SC-CO_2.

The solubility temperature dependence at constant pressure and the resultant natural convection currents are thus exploited to clean the part and regenerate the carbon dioxide within a single vessel. In this fashion, the CO_2 usage is independent of cleaning time (it is based solely on the amount of CO_2 used during parts introduction and removal), and energy usage is less since only heat exchangers are employed without a compressor.

Fluid transfer from the initial contaminant-saturated SCF in the top section to the "clean" SCF in the distillation-like bottom section is effected by a single hole in the center of a baffle. This fluid transfer occurs from a plume of hot fluid "burping" into the cold section, followed by a plume of cold fluid burping into the hot section. This pattern is repeated continually with at least 5 exchanges every 2 seconds until the contaminant concentration in the cold section approaches the concentration in the hot zone (estimated to be 1-2 orders of magnitude lower than the cold saturation concentration, depending on contaminant composition).

The general configuration of the SCF cleaning chamber is shown on Figure 6. The chamber consists of both a horizontally and vertically oriented 11-inch inside diameter cylindrical vessel connected by a cross section. The vertical portion is where the cleaning process occurs, and the horizontal portion contains the transfer chamber and parts basket transportation mechanism and structure. The cleaning and transfer chambers are separated from one another by a ball valve.

The parts to be cleaned are inserted into the cleaning chamber at the cross location through the transfer chamber. A parts basket and a chain-driven rail transport system are used to transport the parts from the transfer chamber into the cleaning chamber. The horizontal extension of the cleaning chamber, opposite the transfer chamber, houses the actual mechanisms used to move the parts basket from the transfer chamber and into and out of the cleaning chamber cross. Clamped connections are used to connect the end closures and ball valve to the chamber.

V. EQUIPMENT

The preproduction cleaner consists of the pressure vessel with its mechanical equipment and connectors, the piping and instrumentation subsystem for process control and carbon dioxide transfer, and the facility to house, operate, and maintain the cleaner. Each major subsystem is discussed in the following sections. Schematics and electrical drawings are presented throughout for enhanced clarity.

A. Pressure Vessel Components

Figure 7 is an exploded view of the preproduction cleaner. Whenever possible, existing commercially-available components were selected to enhance the marketability, reduce cost, and minimize delays associated with "one-of-a-kind" parts.

The pressure vessel was designed and fabricated in accordance with American Society of Mechanical Engineers (ASME) B31.3 Piping Code and ASME Boiler and Pressure Vessel Code, Section VIII, Divisions 1 and 2. The vessel does not carry an ASME stamp, since the vessel will be employed as a research tool and thus it was decided not to hire the ASME inspector.

Pressure vessel lifetimes are determined by their pressurization and depressurization cycles. The transfer chamber is designed for 30,000 pressure cycles while the cleaning chamber is designed for 990 pressure cycles. These numbers were selected to give approximately five years of operation in a production facility cleaning 24 batches of parts every day. During a single cleaning, the transfer chamber goes through one cycle while the cleaner chamber goes through none (operates isobarically). The cleaning chamber goes through a pressure cycle only during maintenance or a system shutdown. Since the parts basket is large (8.25-inch diameter and 12 inches long), many parts can be cleaned simultaneously.

A pressure cycle occurs whenever the highest and lowest pressure swing is greater than 20% of the design pressure (2150 psi). The computer system automatically logs the pressures in the transfer and cleaning chambers and records the high and low pressures of every occurrence exceeding 400 psi differential. Since the pressure cycles of the cleaner rarely encompass the entire 2150 psi range, the lifetime of the vessel can probably be extended by means of a fatigue analysis utilizing the historical pressure cycle data.

1. TubeTurns Closure

The parts access is through the TubeTurns Closure, a steel hatch with hinges. The hatch is secured by two C-clamps (called the "yoke") which are opened and closed with a mechanical screw drive actuated by the computer control system. When the yoke is only slightly open (approximately ½ inch), the steel hatch (referred to as the "door") is retained even if pressure exists in the transfer chamber. This is an added safety feature to prevent rapid decompression. SwRI mounted an emergency stop button near the hatch in the event the yoke begins to close and an object might be crushed. Figure 8 is the wiring diagram for the yoke controls.

2. Twelve-inch Through-bore Ball Valve

The valve which separates the transfer and cleaning chambers is the most expensive, heaviest, and longest lead time component of the entire preproduction cleaner. The valve and electrical

actuator weigh more than 60% of the entire system. After exhaustive valve searches, SwRI purchased the Valvtron ball valve with a 11-inch through-bore for passage of the parts basket with minimal obstacles (grooves or lips on the interior of the pipe, connector, and valve). The carbon steel ball valve with Metco 19 coating was purchased in lieu of a stainless steel body for significant cost reduction. Unfortunately, not all of the wetted surfaces are coated, so corrosion is of concern. Atmospheric moisture can condense inside the cleaner and form carbonic acid in the presence of pressurized carbon dioxide. Dilute solutions of carbonic acid are the most corrosive and will rust the carbon steel. Periodic inspections are advised to monitor the degree of corrosion so that the valve can be taken out of service prior to catastrophic failure.

3. Automated Parts Basket Transport System

The parts basket is a cylinder consisting of two circular solid aluminum end plates and 18 removable rods evenly distributed around the circumference. The basket is designed to rotate, and parts are secured by tying with metal wire onto the rods. The end plates are mounted on wheels for easy movement through the horizontal run of the pressure vessel. The internal basket size is 8.25 inches in diameter and 12 inches long; parts as heavy as 70 pounds can be cleaned.

In the cleaning chamber there is a rail-mounted, chain-driven carriage with a cantilever arm. Figure 9 depicts the parts basket in the pick-up position with the cantilever arm extending through the ball valve. This arm hooks onto the basket end plate, and then the motorized chain drive moves the basket into the cleaning chamber (see Figure 6 for basket in cleaning position). During cleaning, basket rotation is accomplished by engagement of a coupling-half on one end of the parts basket and a coupling-half on the end of a motorized shaft at the back of the pressure vessel horizontal run. When cleaning is complete, the chain drive transports the basket into the transfer chamber and a metal bar depresses the cantilever arm latch release trigger, thereby freeing the basket. The cantilever arm is then pulled back into the cleaning chamber so the ball valve can be closed.

Momentary switches are positioned in the horizontal run of the pressure vessel and are used to indicate the locations of the parts basket and carriage. Actuators for the momentary switches are mounted on the carriage and parts basket. The computer data acquisition system tracks the locations of the carriage and parts basket to ensure successful pickup and release and prevents closing the ball valve on the basket or cantilever arm. Figure 10 is the wiring diagram for these position indicators.

4. Grayloc Connectors and End Hubs

Grayloc connectors were selected to make the cleaner easy to disassemble since the preproduction unit is a first prototype for development purposes and is a research tool. The Grayloc connector weighs less and requires less labor for assembly and disassembly than American National Standards Institute (ANSI) flanges, but cost is a little higher.

5. Thermoelectric Cooler

To cool the top of the cleaner, a thermoelectric cooler was selected instead of a refrigerated chiller using freon. Thus no ODS materials are needed to support the cleaner. The thermoelectric cooler chills recirculating water which is pumped though a stainless steel thick-walled tubing (to prevent collapse during cleaner pressurization) coiled inside the vessel and provides 500W of cooling. Additional cooling occurs through the stainless steel pressure vessel walls to the ambient air.

6. Resistance Heaters

An immersion heater is mounted to the bottom blind hub and raised into the hot zone of the cleaner. The heater is bent to form a 9½-inch cylinder (see Figure 11) so that the heat is concentrated at the outer walls, consistent with the fluid dynamics model. After filling the cleaning chamber with liquid CO_2, initial heating of the system is complimented with externally-mounted band heaters. Half of the band heaters are left on during cleaning so that the immersion heater can provide fine temperature control and keep the hot zone within ±0.5 °C of the set point.

B. Piping and Instrumentation

There are only three types of piping lines to the preproduction unit: (1) cooling water from the thermoelectric cooler, (2) carbon dioxide liquid syphon feed, and (3) carbon dioxide gas purges. The water system is a simple recirculating system including a centrifugal pump, strainer, and cylindrical reservoir for purge capacity and so a schematic was never developed. The carbon dioxide system is more complicated; the following paragraphs describe its configuration.

The carbon dioxide used in the SCF natural convection cleaner is industrial grade liquefied gas supplied in cylinders. The purity of this supply is adequate for the natural convection cleaner since the impurities should phase separate in the hot zone if their concentration exceeds saturation, just like the oils and greases removed from the parts being cleaned.

Figure 12 is the piping diagram for the carbon dioxide system. All of the solenoid valves are operated by the computer system during an automated cleaning cycle or when activated from the system-manager panel. The cylinders are connected to a manifold so that large flow rates of liquid CO_2 can be achieved. The syphon is initiated by simultaneously opening the fill and exhaust valves to the chamber being filled. The exhaust valve is opened during the liquid CO_2 fill so that a slight pressure differential will exist between the chamber and CO_2 cylinders, allowing for continuation of the syphon action. Relatively small quantities of CO_2 are lost through the exhaust valve because of the small orifice. All of the lines are ¼, ⅜, or ½- inch diameter high pressure, stainless steel tubing with Swagelok fittings.

The external band and internal immersion heaters supply sufficient energy to change the liquid CO_2 to SC-CO_2 and raise the pressure to the desired operating condition. The mass of CO_2 in the cleaning chamber controls the pressure for a given temperature. The CO_2 supply cylinders are positioned on an electric scale so that the operator can accurately control the mass of CO_2 added.

The instrumentation package includes thermocouples for temperature control and theoretical model validation, pressure transducers for cleaning sequence control, position switches for the basket transport subsystem control, and TubeTurns yoke control. There is also an imaging system to view the parts basket during cleaning. The instrumentation diagram locating these devices is presented as Figure 13. The wiring diagram for the analog inputs to the computer is Figure 14. Only two local pressure gauges are mounted on the cleaner. These are 6-inch dial, 0-3000 psi gauges facing the operator at the TubeTurns door and indicate the transfer and cleaning chambers' pressures.

The control and data acquisition system computer is an IBM-compatible Pentium 90 MHZ personal computer. Software for the system was developed using the C programing language and high-level functions provided by National Instruments Lab Windows/CVI. The block diagram depicting the relation

between the computer, data acquisition and control cards, and the signal conditioner for minimizing electrical noise is shown in Figure 15.

Two viewports are mounted 180° from each other right below the cross of the cleaning chamber. A 20W lamp shines through one port, and a camera is positioned in the other. Internal mirrors can be positioned to shine the light onto the parts basket and to reflect the basket image into the camera view. The camera output is directly linked to a television with an internal VCR for video recording of the cleaning activity.

Table 2 details the sequences programmed in the automated cleaning cycle. These procedures note the status of instrumentation devices as part of the operational control. Figure 16 is the wiring diagram from the pressure vessel component controls to the control card. The continuous power requirement for the entire cleaning system is 6.5 kW; maximum power usage if every valve, heater, and motor controller is activated is 14 kW. Details of the power requirements are presented in Table 3.

C. Facility Requirements

The preproduction SCF natural convection cleaner is depicted in Figures 17-21 as isometric, top, front, right side, and rear views. The floor space occupied by the cleaner is approximately 6 feet in width and 10.5 feet in length. The length space should be increased by an additional 6 feet for the platform for adding parts to the cleaner. In addition, ample space should be provided on both sides of the cleaner for equipment and instrumentation access. If the chain drive subplate is to be removed from the rear, 6 feet of clear space is required. The subplate may be removed or installed from either end of the horizontal section. The room height should be at least 15 feet from floor to crane hook to allow clearance for mounting the top hub and internal cooling coils.

1. *Work Platform and Stairs*

The preproduction cleaner needs a platform and stairs complete with the OSHA-required railings and kick-plates. SwRI has prepared a blueprint (Drawing No. 6144-000-000) for a recommended working platform; the drawing is part of the Production Drawings documentation package. The system is designed for a maximum of 70-lb payload within the parts basket so consideration for lifting the parts for cleaning onto the platform is needed by the Air Force installation facility.

2. *Electrical Requirements*

The cleaner requires 110-120 VAC, 1-phase, and 208-240 VAC, 1-phase and 3-phase, and the associated breakers as presented in Table 4.

TABLE 2. AUTOMATIC CLEANING CYCLE CONTROL SEQUENCE

SEQUENCE	NORMAL SEQUENCE ACTION	ABORTED SEQUENCE ACTION	ERRORS
Operator initiates cleaning cycle by pressing START CLEAN CYCLE button.	System checks the following parameters before starting a cleaning cycle: door position, basket position, carriage position, system-control voltage, yoke voltage, ball-valve voltage, and limit/position switch voltage. It then acquires cleaning-cycle parameters from front-panel controls. At start of cleaning cycle, the system also disables stripchart updates, menu-bar selections, and all front-panel controls except the ABORT button. Additionally, thermocouple indicators (except T1 and T7) are blacked out because no new values are acquired. Flags used to initiate the first action (Close Yoke) are set to TRUE.	Not Applicable	If errors are detected, the operator is notified via the front-panel message center and the cleaning cycle is not allowed to begin. The following errors prevent start of the cleaning cycle: (1) door is opened, (2) carriage and basket are not in the correct positions, and (3) critical voltages are absent.
Close Yoke	The close-yoke circuit is activated. Close-yoke circuitry is monitored until the yoke has completely closed. After the yoke has closed and no ABORT event has been detected, flags that initiate the next cleaning-cycle sequence (Purge/Fill Transfer Chamber) are set to TRUE.	Any ABORT issued during this sequence is acted upon after the yoke has completed its closing cycle. An ABORT message is displayed for the operator, and the cleaning cycle jumps to the Open Yoke sequence (i.e., those sequences that have been executed are reversed).	The system monitors the elapsed time the close-yoke circuitry is active; if this time exceeds a predetermined value, a time-out error occurs causing immediate termination of the cleaning cycle. Time-out errors can be caused by power failure or electrical/mechanical failure of yoke components.
Purge/Fill Transfer Chamber	The transfer chamber is pressurized and purged according to system parameters (number of pressurization/purge cycles, level of pressurization-psi, level of purge-psi) that are accessible through the system-manager panel. This procedure is used to reduce the volume of atmospheric gases to negligible levels. After completing the specified number of pressurization/purge cycles, a LIQUID CO2 FILL button is displayed over the START CLEAN button; the operator is instructed to record the liquid CO2 weight before pressing the LIQUID CO2 FILL button. A CONTINUE CLEAN button is displayed after the LIQUID CO2 FILL button has been pressed and subsequently removed from the panel; the operator is instructed to press this button after the desired mass of liquid CO2 has been siphoned into the transfer chamber. The flags that initiate the next sequence (Pressure Equalization) are set to TRUE.	An ABORT during the pressurization/purge cycles causes this sequence to halt immediately. The cleaning cycle then jumps to the Purge Transfer Chamber sequence, after which it executes the Open Yoke sequence. The ABORT button is not active while liquid CO2 is being siphoned into the system. Instead, the operator must press the CONTINUE CLEAN button to stop the liquid CO2 fill, after which the ABORT button is reactivated.	No time-out can occur during this part of the cycle; however, if a power failure is detected, the cleaning cycle is immediately halted and no further sequences are executed. After power is restored, the operator can use system-manager controls to reverse those sequences that had been executed.
Pressure Equalization	A solenoid valve that connects the cleaning and transfer chambers is opened so that pressure between the two chambers can be equalized.	An ABORT immediately closes the pressure-equalization solenoid. The cleaning cycle then jumps to the Purge Transfer Chamber sequence, after which it executes the Open Yoke sequence.	No time-out or power failure conditions are monitored. If it is obvious that the chamber pressure are not equalizing, then the operator can ABORT the cleaning cycle.

TABLE 2. AUTOMATIC CLEANING CYCLE CONTROL SEQUENCE (CONT.)

SEQUENCE	NORMAL SEQUENCE ACTION	ABORTED SEQUENCE ACTION	ERRORS
Open Ball Valve	The open-ball-valve circuit is activated. Open-ball-valve circuitry is monitored until the ball valve has completely opened. After the ball valve has opened and no ABORT event has been detected, flags that initiate the next cleaning-cycle sequence (Pick-Up Parts Basket) are set to TRUE.	Any ABORT issued during this sequence is acted upon after the ball valve has completed its opening cycle. An ABORT message is displayed for the operator, and the cleaning cycle jumps to the Close Ball Valve sequence, followed by the Purge Transfer Chamber and Open Yoke sequences.	The system monitors the elapsed time the open-ball-valve circuitry is active. If the elapsed open-ball-valve time exceeds a predetermined value, a time-out error occurs causing immediate termination of the cleaning cycle; operator notification is via the message center. Time-out errors can be caused by power failure or electrical/mechanical failure of ball-valve components.
Pick-Up Parts Basket	During this sequence, the carriage is moved from its cleaning-chamber position to the basket pick-up position. The state of an internal limit/position switch is monitored to determine when the carriage has reached the correct location. If no ABORT signal has been detected, flags that initiate the Move Parts Basket to Cleaning Chamber sequence are set to TRUE.	If an ABORT is issued during movement of the carriage to the basket pick-up position, the carriage drive motor is immediately deactivated, operator notified, and the cleaning cycle advanced to Move Carriage to Cleaning Position sequence. An ABORT issued after the carriage has reached the basket pick-up position, causes the cleaning cycle to jump to the Move Basket to Release Position sequence, followed by Move Carriage to Cleaning Chamber Position. Remaining ABORT sequences include Close Ball Valve, Purge Transfer Chamber, and Open Yoke.	The system monitors the elapsed time the motor has been active during the Pick-Up Parts Basket sequence; if this elapsed time exceeds a predetermined value, a time-out error occurs causing immediate termination of the cleaning cycle. Operator notification is via the message center. Time-out errors can be caused by power failure, limit/position switch failure, motor failure, or mechanical failure of the carriage drive system.
Move Parts Basket to Cleaning	The carriage, with the parts basket attached, is moved to the cleaning chamber position. Again, the state of an internal limit/position switch is monitored to determine when the sequence has been completed. If no ABORT has been detected during this step, the flags that initiate the Elapsed Cleaning Time sequence are set to TRUE.	An ABORT issued during movement of the carriage/basket to the cleaning chamber causes immediate deactivation of the carriage drive motor. The cleaning cycle is advanced to the Move Basket to Release Position sequence, followed by Move Carriage to Cleaning Chamber Position, Close Ball Valve, Purge Transfer Chamber, and Open Yoke.	Errors that can cause immediate termination of the cleaning cycle include: (1) time-out error moving basket/carriage to cleaning-chamber position, (2) basket still at pick-up position after carriage has started moving to cleaning chamber (the system will attempt to correct this problem by jumping back to the Pick-Up Parts Basket sequence; however, if unsuccessful after three attempts, the cleaning cycle is terminated), (3) basket released from carriage during sequence; basket position unknown.

TABLE 2. AUTOMATIC CLEANING CYCLE CONTROL SEQUENCE (CONT.)

SEQUENCE	NORMAL SEQUENCE ACTION	ABORTED SEQUENCE ACTION	ERRORS
Elapsed Cleaning Time (Actual parts cleaning sequence)	During this sequence, the elapsed time that the parts basket is in the cleaning position is monitored; elapsed cleaning time is continually updated and displayed on the main panel. At the start of this sequence, stripchart updates are reactivated, digital temperature readouts for all thermocouples are returned to normal, and the Graph menu-bar selection is made available to the operator. If the data-record switch had been set to the on position, data is recorded to the operator-specified file at operator-specified intervals. At the end of the sequence, flags that initiate the Move Parts Basket to Transfer-Chamber Release sequence are set to TRUE (note these flags are set to TRUE whether an ABORT has been issue or the sequence terminated naturally), aforementioned controls/indicators are again disabled or blacked out, and the ABORT button is disabled because an abort is no longer valid.	If an ABORT is issued, the Elapsed Cleaning Time sequence is terminated, regardless of the amount of elapsed time; if data recording is active, the data file is immediately closed. All remaining sequences are then executed so that the system is returned to a pre-cleaning-cycle condition.	Not applicable
Move Parts Basket to Transfer-Chamber Release	During this sequence, the carriage/basket is moved from the cleaning-chamber position to the basket release position in the transfer chamber. The state of an internal limit/position switch is monitored to determine when the carriage/basket has reached the correct location. Flags that initiate the Move Carriage to Cleaning Chamber sequence are set to TRUE at the end of this sequence.	Not applicable	The system monitors the elapsed time the motor has been active during the Move Parts Basket to Transfer-Chamber Release sequence; if this elapsed time exceeds a predetermined value, a time-out error occurs causing immediate termination of the cleaning cycle. A time-out error can be caused by power failure, limit/position switch failure, motor failure, or mechanical failure of the carriage drive system.
Move Carriage to Cleaning Chamber	The carriage is moved to the cleaning chamber position during this sequence; an internal limit/position switch is monitored to determine when the carriage has reached its proper position. After successful completion of this sequence, flags used to initiate the Close Ball Valve sequence are set to TRUE.	Not applicable	If the elapsed time the motor has been active during the Move Carriage to Cleaning Chamber sequence exceeds a predetermined value, a time-out error occurs causing immediate termination of the cleaning cycle. Additionally, the system checks to insure proper basket. If the basket did not release, up to three attempts will be made to correct the error (previous sequence rerun); if not corrected, the cleaning cycle is terminated and no subsequent sequences performed.

TABLE 2. AUTOMATIC CLEANING CYCLE CONTROL SEQUENCE (CONT.)

SEQUENCE	NORMAL SEQUENCE ACTION	ABORTED SEQUENCE ACTION	ERRORS
Close Ball Valve	The close-ball-valve circuit is activated. Close-ball-valve circuitry is monitored until the ball valve has completely closed. After the ball valve has closed, flags that initiate the next cleaning-cycle sequence (Purge Transfer Chamber) are set to TRUE.	Not applicable	The system monitors the elapsed time the close-ball-valve circuitry is active, and if it exceeds a predetermined value, a time-out error occurs causing immediate termination of the cleaning cycle; no subsequent steps are executed. Time-out errors can be caused by power failure or electrical/mechanical failure of ball-valve components.
Purge Transfer Chamber	The transfer chamber purge valve is activated; this valve remains open until the transfer-chamber pressure is at or near atmospheric pressure. The ABORT button is reactivated for this sequence so that if the valve malfunctions or if a power failure occurs, the operator can terminate the cycle. After completion, flags used to initiate the Open Yoke sequence are set to TRUE.	An ABORT can be issued by the operator during this sequence; however, it simply terminates the cleaning cycle and prevents the last sequence from being performed.	See ABORT SEQUENCE ACTION
Open Yoke	The open-yoke circuit is activated. Open-yoke circuitry is monitored until the yoke has completely opened. After the yoke has opened, the operator is notified that the parts basket can be removed and the system is returned to normal operation (all control/indicators and menu-bar selections reactivated).	Not applicable	The system monitors the elapsed time the open-yoke circuitry is active; if this time exceeds a predetermined value, a time-out error occurs causing immediate termination of the final sequence. Time-out errors can be caused by power failure or electrical/mechanical failure of yoke components.

TABLE 3. POWER REQUIREMENT DETAILS FOR PREPARATION DEVICE

Component	Voltage	Rated Current	Power
Carriage Motor w/ Gearhead, Oriental Motor 3RK15GN-AUL/3GN120KA	115VAC	0.36A	41.4W
Basket-Rotation Motor w/Gearhead, Oriental Motor 61K60GK-AUL/6GK180K	115 VAC	1.0	115W
Pressure-Transducer Power Supply, Sensotec 28 VDC, 80 mA	120VAC	0.020A	2.4W*
Signal-Conditioner Power Supply, Analog Devices # 955, 5VDC, 1.0A	120VAC	0.1A	12W*
Cleaning-Chamber Purge Solenoid Valve, Gold Ring Model CFGC05	120VAC	0.092A	11W
Cleaning-Chamber Purge Solenoid Valve, Gold Ring Model CFGC05	120VAC	0.092A	11W
Contaminant-Removal Solenoid Valve	120VAC	0.4A	48W
Transfer-Chamber Purge Solenoid Valve	120VAC	0.4A	48W
Pressure-Equalization Solenoid Valve	120VAC	0.4A	48W
Cleaning-Chamber 2-Way Ball Valve, Whitey	120VAC	1.7A	204W
Transfer-Chamber 2-Way Ball Valve, Whitey	120VAC	1.7A	204W
Cooling System Pump, Teel Cast Aluminum Centrifugal Pump	120VAC	8.2A	984W*
Thermoelectric Cooler	240VAC	5.3A	1272W*
Internal Immersion Heaters, (2 - 4300W @ 220VAC units are connected in series)	220VAC	8.8A	1936W*
External Band Heaters (3 - 500W @ 120V)	120VAC	12.5A	1500W
External Band Heaters (3 - 500W @ 120V)	120VAC	12.5A	1500W*
External Band Heater (1 - 500W @ 120V Transfer Chamber)	120VAC	4.2A	500W
Yoke Motor	230VAC	6.4A	1472W
Ball-Valve Actuator Motor	230VAC	7.2A	1656W
Lift Table, Autoquip Series 35, Model 48S25	240VAC	5.3A	1272W
Computer (Pentium 90 w/ 250W power supply - assume 70% efficient)	120VAC	3.0A	360W*
Computer Monitor, KFC P/N CA1728, S/N A8KKU4985265	110VAC	1.8A	198W*
TV/VCR Model PV-M2024, S/N E4AA15387	120VAC	0.93A	112W*
Camera	120VAC	0.33A	40W*
Light Source, Radio Shack, Power Supply, Cat. # 22-12013	120VAC	0.5A	60W*
Scale, Model AD-5000	110VAC	3A	330W*

* Denotes those components that are continuously operational. Other components operate sporadically and only for very short durations (in most cases, one minute or less).

MAXIMUM SYSTEM POWER REQUIREMENTS (ALL COMPONENTS)	13.94kW
MAXIMUM CONTINUOUS POWER REQUIREMENTS	6.5 kW

TABLE 4. ELECTRICAL REQUIREMENTS FOR PREPRODUCTION DEVICE

Breaker	Rating	Purpose
1	20A, 208-240VAC, 3-Phase	Yoke motor and associated control circuitry
2	20A, 208-240VAC, 3-Phase	Ball-valve actuator motor and associated circuitry
3	20A, 208-240VAC, 3-Phase	Lift table
4	20A, 208-240VAC, 1-Phase	Internal heaters and thermoelectric cooler
5	20A, 110-120VAC, 1-Phase	Power to all control circuitry for the SCF including 5VDC and 28VDC power supplies which feed signal-conditioning modules and pressure transducers, basket travel and rotation motors, and 110-120VAC feed from all master control relays. It is important that a single breaker feed all of the aforementioned components because this voltage is monitored by the program; most system functions check for this voltage before being performed.
6	20A, 110-120VAC, 1-Phase	Duplex outlet for TV camera and cooling system pump
7	15A, 110-120VAC, 1-Phase	Duplex outlet for computer and monitor
8	20A, 110-120VAC, 1-Phase	Outlet for three (3) 500-Watt band heaters
9	20A, 110-120VAC, 1-Phase	Outlet for three (3) 500-Watt band heaters

3. *Material Handling Equipment and Specialized Tools*

A 5-ton overhead crane is required to assemble the preproduction cleaner. The overall gross weight is approximately 6 tons but will be erected from several component pieces. Smaller lifting devices may be desired since the hubs weigh approximately 550 lbs.

A small forklift is recommended to move the carbon dioxide gas cylinder pallets during weighing operations to quantify carbon dioxide usage and to prevent overfilling of the cleaning or transfer chambers. (Overfilling safety measures have been incorporated as evidenced by the pressure relief valves and rupture discs.)

The only specialized tool required is a pneumatic wrench to tighten the four bolts on each Grayloc connector. Each stud is 1¾-inch with a pitch of 8 threads per inch (SA 193 1¾"-8UN-2A) and needs an average torque of 870 ft-lb.

4. *Unique Safety Requirements*

Standard industrial safety practices should always be employed when working with the preproduction device. All pressure vessel code requirements for pressure relief valves, rupture discs, mechanical strengths of materials, etc. have been incorporated. Numerous safety and hazard reviews have occurred, and the most recent detailed one was prompted by a fatality report[19] from a small biotechnology company doing SC-CO_2 extraction; SwRI was already practicing the recommendations from the report and found no faults which might lead to a similar accident with the preproduction unit. Of significant note, the TubeTurns closure used on the preproduction cleaner is inherently safer than the screw cap used by the small company. The TubeTurns closure has an internal pressure differential switch and a computer interlock based on the transfer chamber pressure transducer reading to prevent opening when pressure is above 3-5 psig inside the transfer chamber. If these are defeated, the large C-clamps will keep the door from flying open from a sudden pressure release so that venting can occur around the closure facing.

An oxygen monitor is advised in the work space to warn about decreasing oxygen levels in the event of a carbon dioxide leak. In addition, a good ventilation system should be provided to prevent worker health hazards from the same source.

Pressure containment (e.g., a polycarbonate shield) is not necessary since the pressure vessel has been constructed and inspected in accordance with ASME pressure vessel codes (refer to "Design Report for Non-Ozone Depleting Supercritical Cleaning Fluids Chamber").[20]

5. *Carbon Dioxide Source*

The entire cleaning system (transfer chamber and cleaning chamber) has a volume of 315 liters. At room temperature (22 °C), liquid carbon dioxide has a density of 0.738 g/cc. At the critical point, SC-CO_2 has a density of 0.466 g/cc. The preproduction cleaner is designed for a maximum pressure of 2,150 psig. As long as the operating temperatures at the highest pressure exceed 37 °C in the cold zone and 47 °C in the hot zone, pumps are not required, and the cleaning device requires less than 10 cylinders (each cylinder nominal contents are 22 kg mass) of liquid carbon dioxide to fill it via liquid syphon.

During operation, only the transfer chamber is purged and filled with carbon dioxide (volume of 62 liters or about 20% of the entire chamber). Thus, no more than two liquid carbon dioxide cylinders (costing about $5) will be needed for each cleaning batch.

SwRI will supply a scale and two gas cylinder pallets (4 cylinder capacity for each) designed for forklift handling. Sufficient floor space is needed to store these pallets (32-in by 22-in each) and the scale (24-in by 24-in). In addition, gas cylinder supply should be readily available.

VI. DESIGN PROCEDURES AND OPERATING MANUAL

SwRI formed a design team consisting of (1) mechanical designers and engineers knowledgeable in pressure vessels, fabrication, automation, and vision systems, (2) an electrical engineer and electronics technicians with expertise in data acquisition, process control, and field installations, (3) computational fluid dynamicists with natural convection experience, and (4) chemical engineers familiar with chemical phase equilibrium and supercritical fluids. This design team held regular meetings to debate critical design decisions and to review status on different task assignments. The design meeting frequency depended upon project activity and accelerated to weekly gatherings about two months before procurement commenced. Individual members frequently met informally, one-on-one to resolve integration issues; this was greatly facilitated since SwRI maintains a single facility and travel was not required.

Regular contact was maintained with the Aircraft Modification Directorate personnel at WPAFB to ensure that their production requirements were met by the cleaning chamber design since they were originally planned to be the end-user. Design review meetings were also held with both USAF representatives from Wright Laboratories and the Aircraft Modification Directorate; these design reviews were responsible for several cost-saving and schedule reduction decisions.

Members of the SwRI design team were empowered to make all decisions on their respective assignment providing the specifications (including compatible interface with other subsystems), budget, and schedule could be met. Issues which had major impact on operability, budget, or schedule were discussed with the project manager to review alternatives before a decision was made with concurrence from the project manager and occasionally with USAF technical representatives. Experts were periodically consulted whenever a technical hurdle was uncovered which the design team was unable to resolve. An example of an expert included a certified industrial hygienist (CIH) who could advise on worker health and safety aspects to ensure that the preproduction unit would meet safety codes as well as the recommended guidelines. These experts were full-time SwRI employees also residing in San Antonio, but their time was not required in sufficient numbers to add them to the design team.

Procurement milestones served as design freeze dates for various subsystems and interface specifications. Design changes after these milestones were permitted if (1) a less expensive route was identified which had no detrimental schedule or performance impact, (2) a given design was later deemed inoperable and required a fix, or (3) safety improvements were found.

Detailed operating instructions for the cleaner were prepared under another contract with the USAF (subcontract through LAI); the October 17, 1995 revision is presented in Appendix B. The instructions are periodically updated to reflect system modifications and lessons learned. They are presented herein as a concise description for how cleaning steps are done and of the computer control system which was designed and implemented under the UDRI subcontract.

VII. RESULTS AND DISCUSSION

Four aspects of this development project are discussed in more detail in the following pages. These issues are (i) patent status, (ii) benefits of multidisciplinary team, (iii) flexibility of preproduction device, and (iv) trade-off assessments for future cleaners.

A. Patent Status

SwRI applied for patent protection on the SCF natural convection device on June 30, 1992 and received U.S. Patent 5,401,322 on March 28, 1995, assigned to SwRI with royalty-free governmental use. This is the base process patent. The original patent application was ruled to contain multiple inventions and, thus, SwRI is filing the others sequentially. Continuations in part will also be filed to protect as many aspects of this technology as possible.

B. Benefits of the Multidisciplinary Team

As described in the credits and acknowledgments section as well as in the procedures section of this report, SwRI relied on numerous technical people with a varied skill base to design and build the preproduction device. The team succeeded with a device which could be fabricated, which incorporated many commercially-available components, and which is successfully cleaning parts.

Two significant contributions from the multidisciplinary team were (1) the need for internal baffles and (2) a single valve to separate the cleaning chamber from the parts loading area. The baffles resulted from a disappointing review of experimental data from the prototype device: every condition cleaned the part of soluble oils, even when natural convection should not have been present (i.e., the hot and cold heat exchangers were controlled to the same temperature).

Computational fluid dynamicists studied the prototype vessel and discovered that the unusually low viscosity and significant density change of the SCF were causing extremely fast convection currents. In fact, temperature differentials as small as 1.5 °C created SCF flow rates as high as 1.8m/sec. Under these conditions, heat transfer into the SCF could not occur: the hotter the heat transfer surface, the faster the SCF flowed. The fluid dynamicists devised baffles (horizontal trays) to slow down the SCF and to create distinct hot and cold zones. Flow patterns can be controlled by selecting appropriate baffle hole patterns and heater locations; these selections affect the mass transfer rates between the hot and cold zones.

The second major result of the multidisciplinary team was the cleaner configuration. At the onset of this subcontract, SwRI was adding parts directly to the cleaning chamber. The cleaning chamber was segregated from the pressurized hot and cold zones using two valves mounted in the vertical column. Figure 2 of the prototype device shows the valve separating the cold zone from the cleaning chamber. When this scheme was scaled from the 2-inch diameter pipe of the prototype to the 12-inch diameter pipe of the preproduction unit, major design problems were realized. These problems were cost, space, weight, bi-directional seals, heating requirements, and long delivery times. During a design review with WPAFB personnel and the SwRI team, alternatives to solve these difficulties were discussed. The solution of a single valve, mounted horizontally and separating the cleaning chamber from a transfer chamber became obvious. The change in perspective was needed to find this simplifying answer.

C. Flexibility of Preproduction Device

The four-month delivery time for the large ball valve and TubeTurns closure necessitated ordering these components before the design could be completed with the CFD input. In addition, the preproduction device is an experimental tool for cleaning trials and so field modifications will be desired. Consequently, nozzles (penetrations with threaded ends) were added on the major components as well as throughout the cleaning chamber piping to accommodate future needs for thermocouples, pressure transducers, electrical connections, sampling devices, fiber optics, etc. Also, the Grayloc connectors were added to the back, top, and bottom segments of the cleaner for maintenance ease, additions of pipe length, and reconfigurations. These decisions have proven wise. The system debugging has repeatedly used several of the nozzles and all of the Graylocs. The request by Lockheed-Martin to clean satellite heat pipes needs cleaning chambers as tall as 5 to 8 feet which is easy to accommodate with more vertical pipe. The new work to design a smart cleaner (one which can tell the operator when cleaning is finished) requires sampling ports which already exist in the hot and cold zones.

D. Trade-off Assessments for Future Cleaners

The natural convection SCF cleaner has many variables which permit an unlimited number of configurations. Future design decisions must evaluate each of the variables (cleaning cycle times, production through-put, cleaning chamber size, need for a transfer chamber and basket transport system) to optimize the device for its intended application. Obviously, these decisions will affect the initial capital investment, operating cost, system complexity, and construction time. The details of these decisions are given in the following paragraphs.

The initial objective of the design was to clean parts in the SC-CO_2 natural convection device in 15 minutes, consistent with the 1,1,1-trichloroethane and Freon 113 vapor degreasers being removed from production facilities. This can be done with the basket transport system operating in 5 minutes and cleaning dwell times in 10 minutes.

During the automated cleaning cycle, the SCF preproduction system can close the yoke, open the ball valve, pick up the parts basket, move it to the cleaning chamber, move it back to the transfer chamber, close the ball valve, and close the yoke all in less than 5 minutes. The purge-fill sequence and decompression steps take additional time. Unfortunately, significant cooling (below 0 °C) occurs from rapid purge-fill sequences causing water condensation and vessel wall stresses, possibly reducing vessel life. When these cold temperatures occur, moisture from the air condenses in the transfer chamber and cannot be removed, creating a severe corrosion concern since a weak carbonic acid solution will form in the presence of carbon dioxide. There are two ways to compensate for the cooling phenomenon: (1) add heaters at the depressurization valves or nozzles, or (2) slow down depressurization to allow heat transfer from the vessel and CO_2 to reach thermal equilibrium. The first requires more energy and controls to operate the system; the second increases the cleaning cycle time. Obviously, the choice between these two options is application dependent.

The actual dwell time for a part in the cleaning zone is dependent upon two variables: (1) the solubility and quantity of the contaminant, and (2) the mass transfer rate of the contaminant from the cold zone to the hot zone. The computational fluid dynamics modeling done under the LAI subcontract showed that with two orders of magnitude difference in saturation concentrations between the hot and cold zones, the mass transfer of 99% of the contaminants could be accomplished in times ranging from 10 minutes to

one hour depending on the baffle configuration. Different baffle designs yield different fluid flow patterns and temperature differentials between the hot and cold zones, so baffle selection is quite complex and depends upon the cleaning application.

Other future design considerations can focus on the possible elimination of the ball valve, transfer chamber, and basket transport system. Currently, approximately \$5 of CO_2 is expended for every load cleaned in the parts basket. If the cleaning vessel was depressurized for every load and the three components were eliminated, then a single load would use about \$15 of CO_2; however, the cleaning times would be significantly longer since the entire system would need to be heated and then equilibrium established between the hot and cold zones. The cleaning chamber wall thickness would need to be slightly increased to give longer usable lifetimes. The most expensive component, the 11-inch through-bore ball valve, would be eliminated. System reliability would be increased since the basket transport mechanism, the only regularly moving parts within the pressure vessel, wouldn't be needed. The trade-off decision would primarily involve the cleaning time -- short cleaning times can be accomplished only with the transfer chamber since establishment of the hot and cold zones can take several hours.

VIII. LESSONS LEARNED

Building and operating the preproduction device has given SwRI tremendous experience which will permit streamlining future natural convection SCF cleaner development efforts. Numerous improvements and simplifications are readily apparent when observing the preproduction device which were either difficult or impossible to envision during the design stage. Many of the ideas for the future vessels are presented in the recommendations section of this report. Following are a few of the lessons learned from the construction of the preproduction device.

Materials selection for SC-CO_2 applications is a difficult task since very little published data exists (i.e., materials compatibility charts do not include SC-CO_2). The *Encyclopedia of Polymer Science and Engineering* does give two lists of soluble and insoluble polymers in SC-CO_2, but several common materials used for seals, coatings, or adhesives are not included. Material selection is further complicated since the co-existence of liquid and gas phases of CO_2 can cause cavitation problems and extreme temperature excursions. Many equipment suppliers have experience with SC-CO_2 and can advise in seal selection, but other suppliers don't understand the difficulties and will supply unacceptable materials.

The pressure vessel is stainless steel selected for corrosion-resistance. The carbonic acid formed when water contaminates the carbon dioxide is highly corrosive as a dilute solution in CO_2. It is virtually impossible to prevent water from entering the clean, especially since many water-soluble lubricants are being introduced in the production shops under solvent substitution efforts.

The first internal baffle was machined from polycarbonate. The heat from the external band heaters during an upset condition and multiple phase transitions among gas, liquid, and SCF CO_2 damaged this baffle. The polycarbonate was "foamed" or expanded, badly warped, and discolored (from clear to yellowish-white). The baffle is now made from Teflon.

The Grayloc connector system made assembly easier and less costly. The device weight was dramatically reduced and size less forbidding for commercial applications than had flanges been used.

Lab Windows/CVI software was an excellent choice for the data collection, operational software, and system controls. During the debugging stages, the software was easily updated for additional safety enhancements and backup checks on the operator. Trouble-shooting of mechanical system failures was eased by the control options of the software. The computer screens were optimized for readability and functionality, and password protects of sensitive operational parameters allowed extra safety security.

The TubeTurns closure has proven highly reliable with mechanical safety features which will minimize catastrophic failures such as the recently-reported fatality at a Massachusetts biotechnology firm employing supercritical fluids. The pressure switch on the closure, however, had a Buna-N diaphragm which failed under the SC-CO_2 conditions and was replaced with a Teflon diaphragm.

To replace all needs for pumps, the CO_2 is provided via syphon fill from compressed gas cylinders. This system has proven reliable, but the cold temperatures from the initial rapid expansion of the liquid created start-up problems including water condensation in the transfer chamber during the purging sequence.

IX. RECOMMENDATIONS

The natural convection SCF cleaner works on a production scale as a result of this subcontract and the complimentary one from LAI. Technology transfer activities have begun and commercial interests have been found. It is imperative to continue the developmental efforts and aggressively pursue commercialization to prevent the natural convection technology from dying on the shelf as an interesting research study. The following seven recommendations are intended as a guideline for the technology development efforts needed to make this technology a commercial success.

A. Collect More Cleaning Data for Degreasing

The experimental studies to date with the preproduction device have shown that mineral oil can be cleaned from the surfaces of metal parts. Under the LAI subcontract, additional testing will be done to study several parts with non-line-of-sight surfaces, different materials, and varying contaminants from the aerospace community. Solubility and co-solvent data will also be obtained under the current funding.

Additional funds will be needed to expand the range of test conditions so that cleaning guidelines can be adopted. These guidelines should include recommended operating conditions for different contaminants based upon solubility data so that an operator could a priori select an operating pressure, hot zone and cold zone temperatures, cleaning dwell time, and co-solvent (if needed). The expanded data set of degreasing experiments will also indicate which contaminants, part shapes, and cleanliness standards are either optimal or won't work for the natural convection SCF cleaner.

B. Proceed with Commercialization Opportunities

Two major equipment vendors of degreasers would like to add the natural convection SCF cleaner to their line of technologies if the cleaning data meet their objectives. In addition, an SCF consultant would like to market the natural convection technology for the dry-cleaning industry. Several USAF depot operations would like to assess the technology for their difficult cleaning applications where ODS materials are still required. Also, the aerospace industry is sending parts for evaluation to SwRI to determine whether specific cleaning tasks can use this new technology.

With this tremendous interest, an aggressive pursuit is needed to find at least one commercially successful application. This first case study will then be the benchmark for future implementation efforts. SwRI management is discussing options and will develop a plan for granting exclusive application licenses. Additional technical data will be needed to determine which fields of use will be economical compared to existing technologies. These data include operating and capital cost needs based upon trade-off design decisions. The economic study must encompass comparisons with organic solvent cleaning, aqueous cleaners, and flow-through SCF devices and contain a life cycle cost assessment for solvent purchase, waste disposal, energy, and depreciation. If possible, an estimate of the capital investment reduction with increasing technology maturation for the natural convection SCF device and comparison of degrees of maturity of the other technology would be beneficial.

Each field of use will have unique demands on the natural convection SCF cleaner and each will require experimental data to confirm cleanliness acceptability. For example, dry cleaning applications of SCF need to be adaptable to small businesses with no chemistry knowledge. Capital investment costs need to be very low, so the ball valve and transfer chamber cannot be included. The substitution of the SCF

device will replace the great dependence on chemical solvents in this industry which currently has minimal choices other than the use of highly flammable solvents like stoddard solvent (100-110 °F flash point).

C. Assemble, Install, Operate, and Optimize "Smart" Cleaner

Under the LAI subcontract, a "smart" cleaner is being developed. The system will periodically sample the hot and cold zone fluids and determine whether equilibrium concentrations have been established. In addition, the system will determine when mass transfer of the contaminant to the hot zone is essentially complete. Under current funding, the goal is to integrate these data into the computer control system and make cleaning dwell time an automatic condition based upon the sampled concentrations. This step should be completed since it will eliminate the need for operator determination of "how clean is clean."

D. Finalize the CFD Model for Future Design Decisions

Under the LAI subcontract, a CFD computer model of the pressure vessel was finished which accounts for the heat and mass transfer in the SCF. The model was used to confirm the design of the baffle built under the UDRI subcontract and described in this report. Additional work under the LAI subcontract was the experimental confirmation and final modifications to the computer code. Consequently, a validated computer model will be available. This model should be used for designing future vessels to confirm that the vessel geometry, cold and hot heat exchanger sizes, and baffle style and location give the needed temperature differentials and SCF flow patterns. The model provides an excellent check on the energy balance calculations to verify the heat exchanger specifications.

E. Revise Heating and Cooling System

Initial energy balances for the preproduction device determined only a 500 W requirement during steady state conditions for the heaters and cooler. The actual operation of the cleaner requires about seven times this requirement, and thus, the thermoelectric cooler is undersized. The extra cooling is occurring by conduction through the stainless steel cold zone vessel walls. Based upon this experience, it may be possible to reduce overall energy needs by having forced convection cooling on the outside wall of the entire cold zone.

The external band heaters on the hot zone walls appear to be more effective and provide better control than the internal immersion heaters. The band heaters are also much less expensive and easier to service; however, localized hot spots may stress the stainless steel vessel wall. A jacket for heating might be a preferred method for precise temperature control of the hot zone wall temperature.

The immersion heaters are controlled from a thermocouple immersed in the SCF and compared by the computer with the operator-selected setpoint. The controls on the cooler have never been used since the thermoelectric cooler is undersized. The CFD computer model should be used with future designs to determine the best control scheme for maintaining large temperature differentials between the hot and cold zones. It is recommended that the heating and cooling designs be re-evaluated in light of the experience gained from operating the preproduction device.

F. Improve Vision System

The cleaning zone is monitored by a camera and images can be video-recorded (fiber optics was not used since the field of view was too small). The current system relies on mirrors to shine light to the parts basket and to reflect the image into the camera. The current design of the mirrors and adjustment device makes fine adjustments difficult and the seals have not been reliable and have periodically leaked. The location of the camera and need for mirrors should be studied on future devices since designs could possibly put the camera in the line of sight of the parts basket. If a transfer chamber is used in the future, then a camera in that chamber would be valuable for noting the condition of the parts during pressurization and decompression.

G. Train Operators and Install at Production Facility

SwRI recommends that significantly more testing of the preproduction device be done at its facilities so that the researchers and designers are readily available for trouble-shooting and interpretation of the findings. Once a cleaning guidance document is prepared (assuming adequate testing can be funded), then the preproduction device will be ready for transfer to a production facility for daily operation. The operators from the production facility will need to be trained by SwRI. It is recommended that SwRI install the device at the production facility. SwRI personnel should also periodically visit the production shop to discuss its acceptance and to make field improvements to the controls or mechanical equipment for easier use.

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APPENDIX A

FIGURES

A

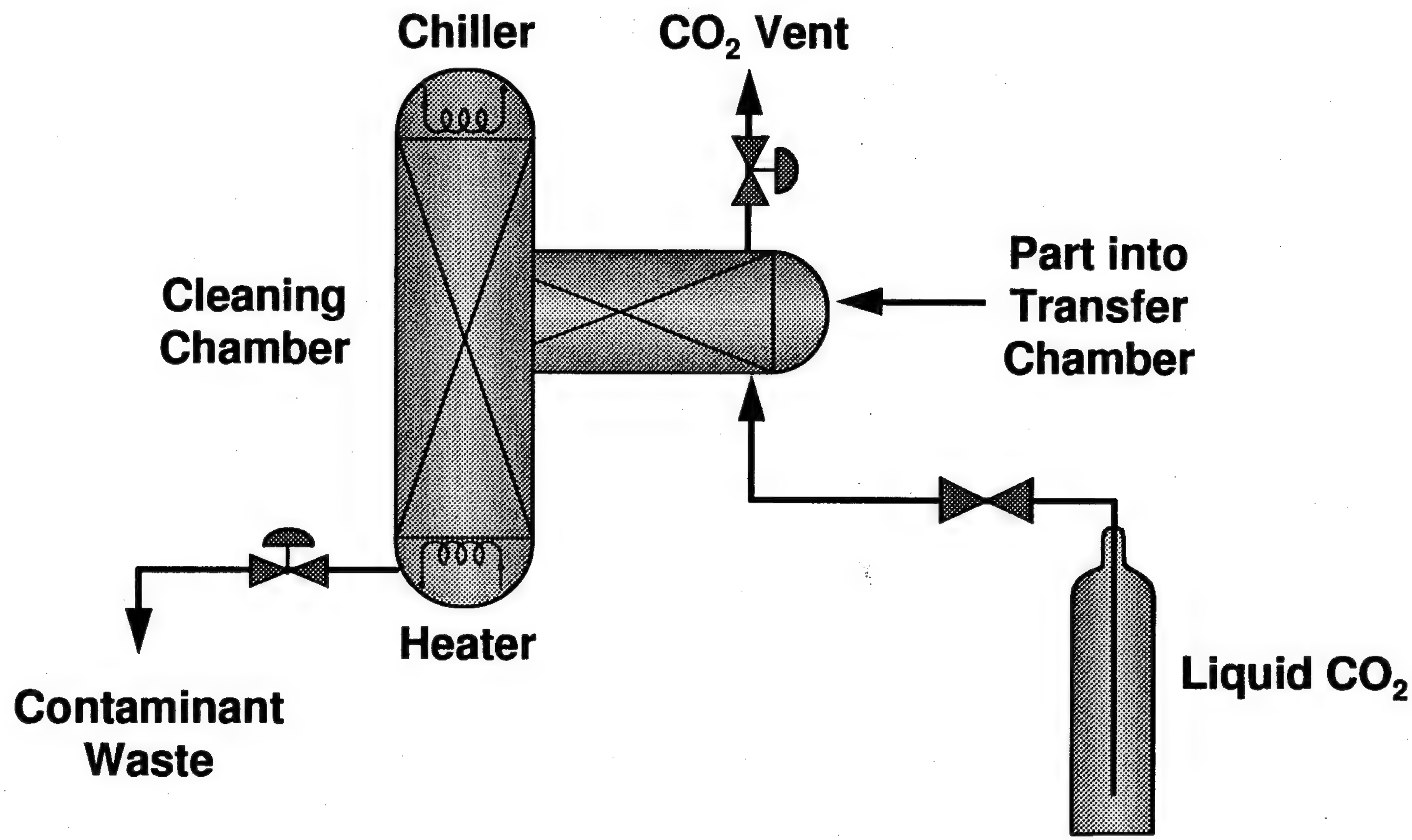


Figure 1. Process Schematic of Natural Convection SC-CO_2 Cleaner

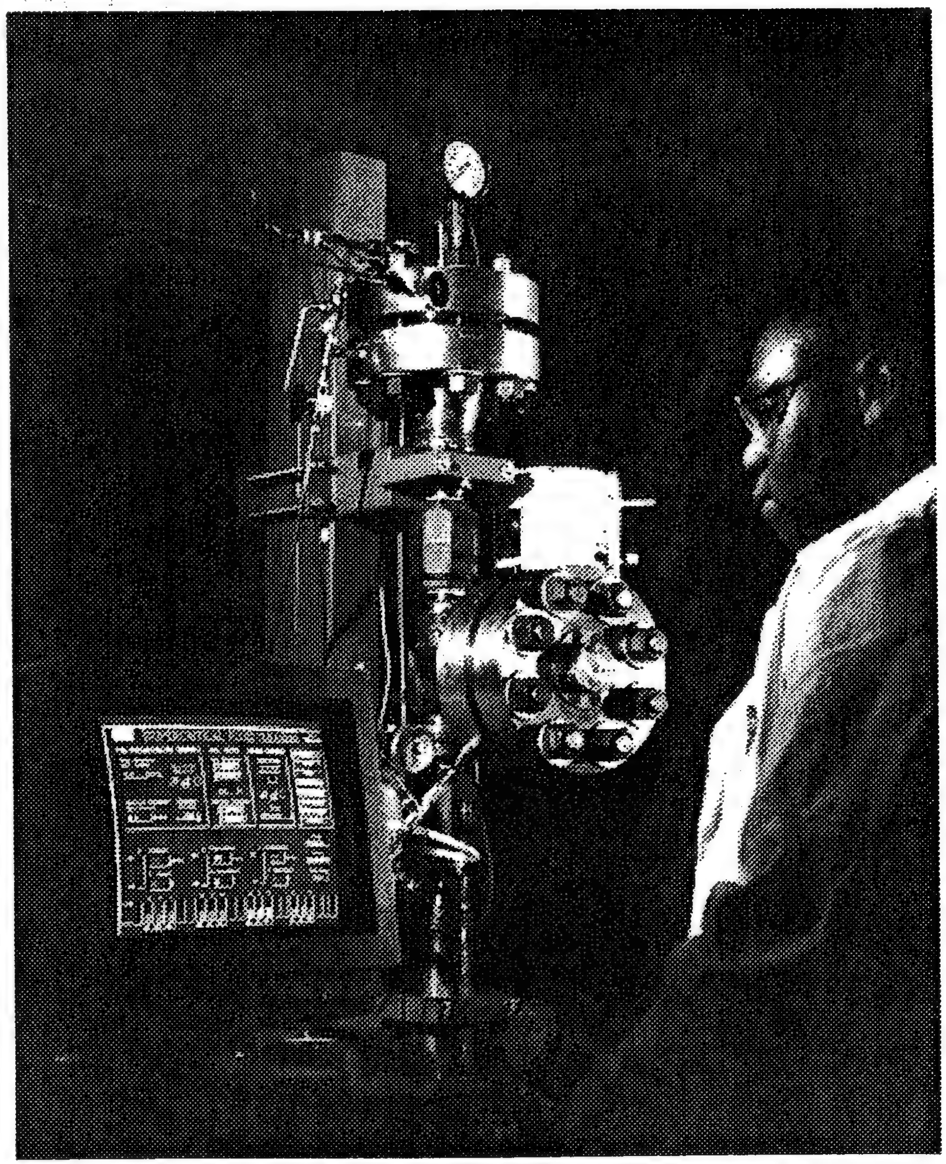


Figure 2. SwRI IR&D Prototype Natural Convection Cleaner

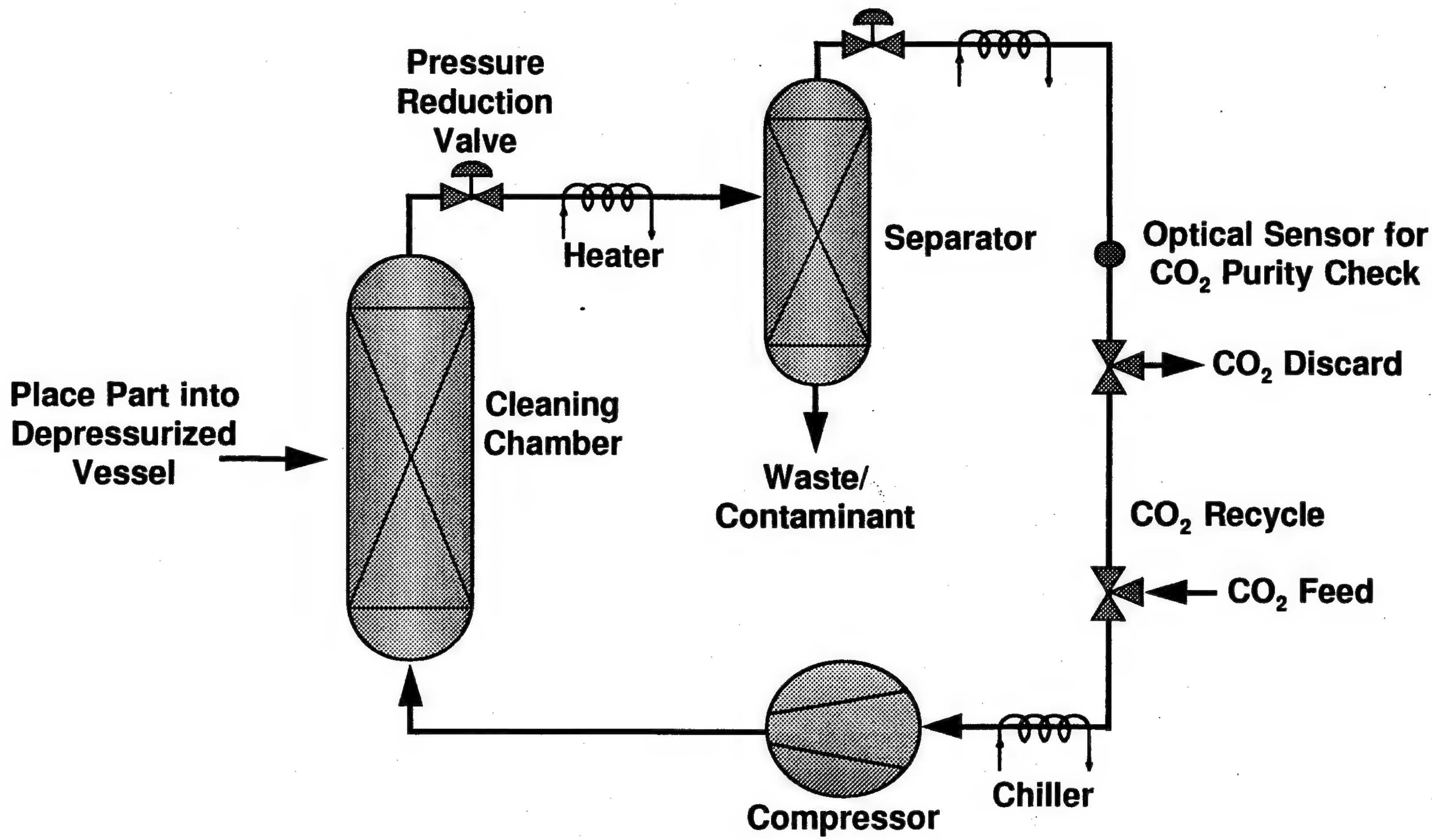


Figure 3. Flow-Through SCF Cleaner Process Diagram

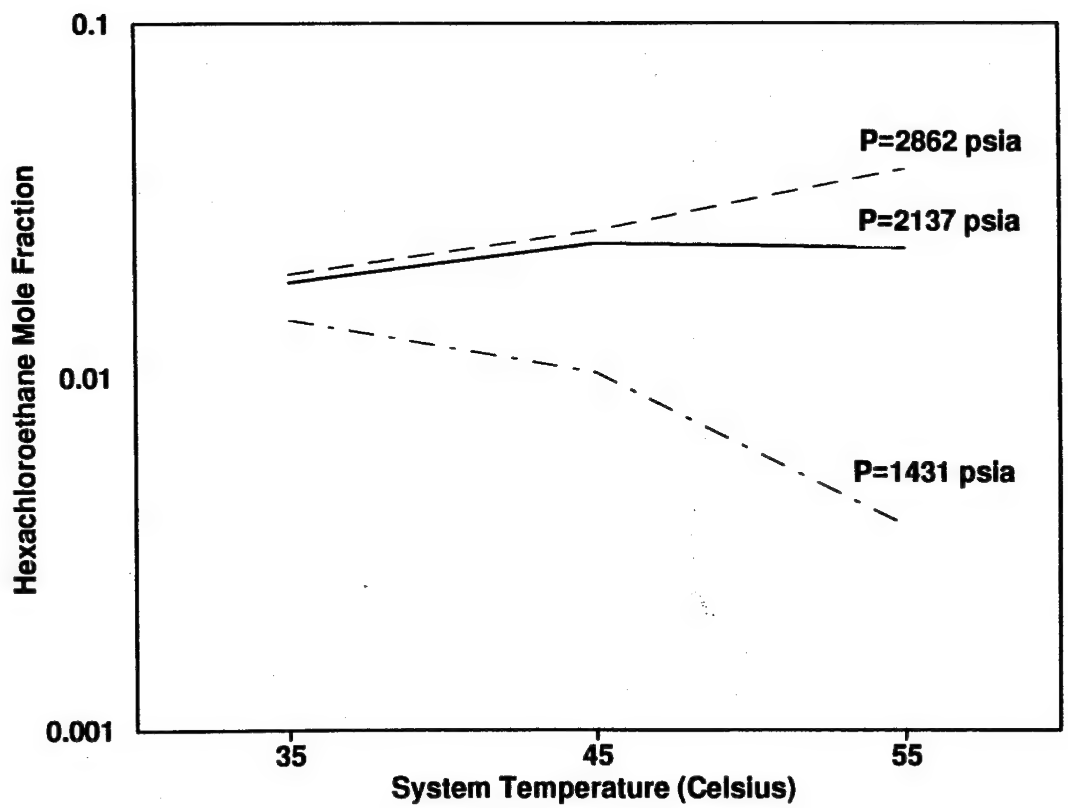


Figure 4. Effect of Pressure and Temperature on Solute Saturated Concentrations

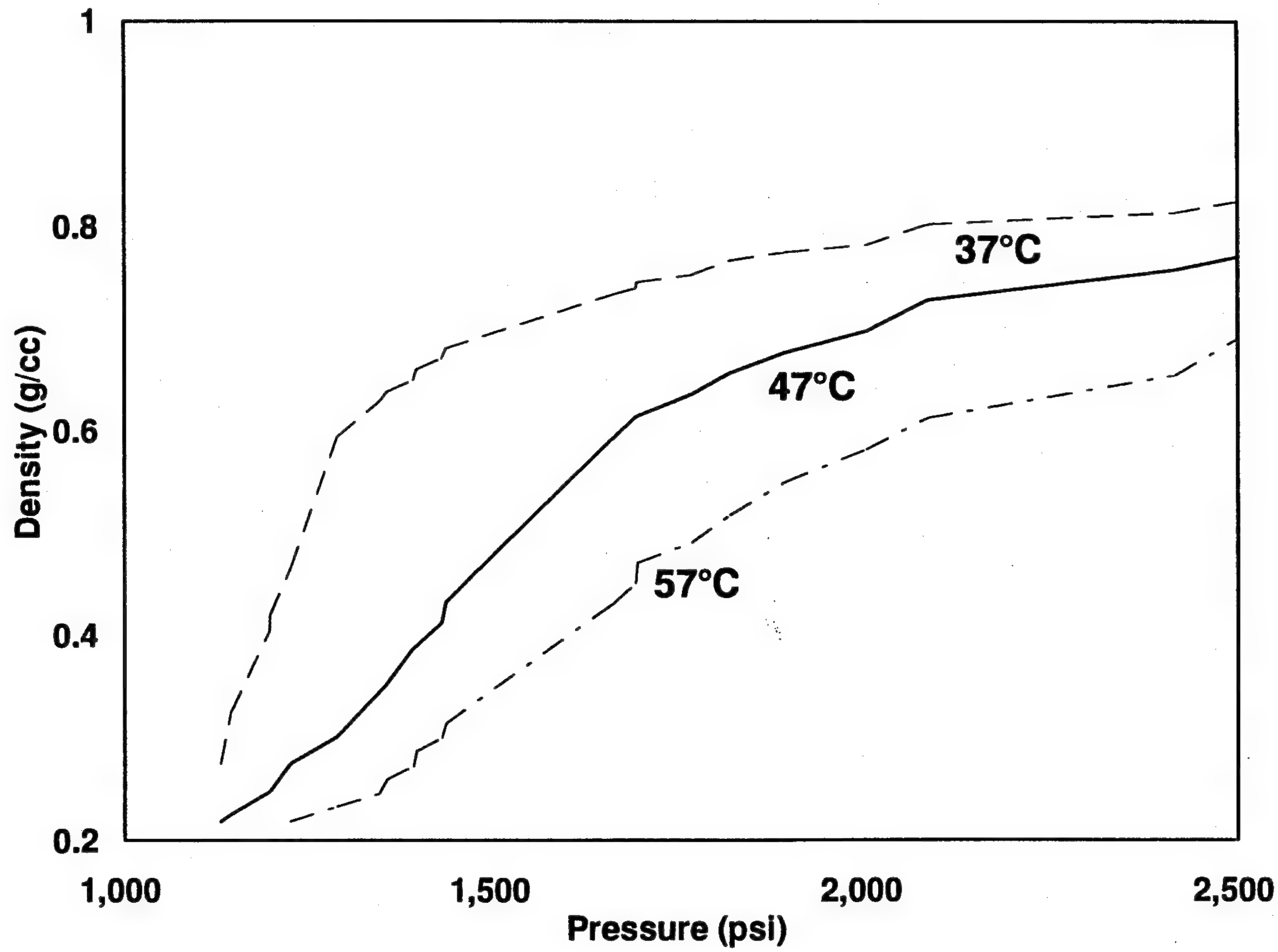


Figure 5. SC-CO_2 Density Versus Pressure Data

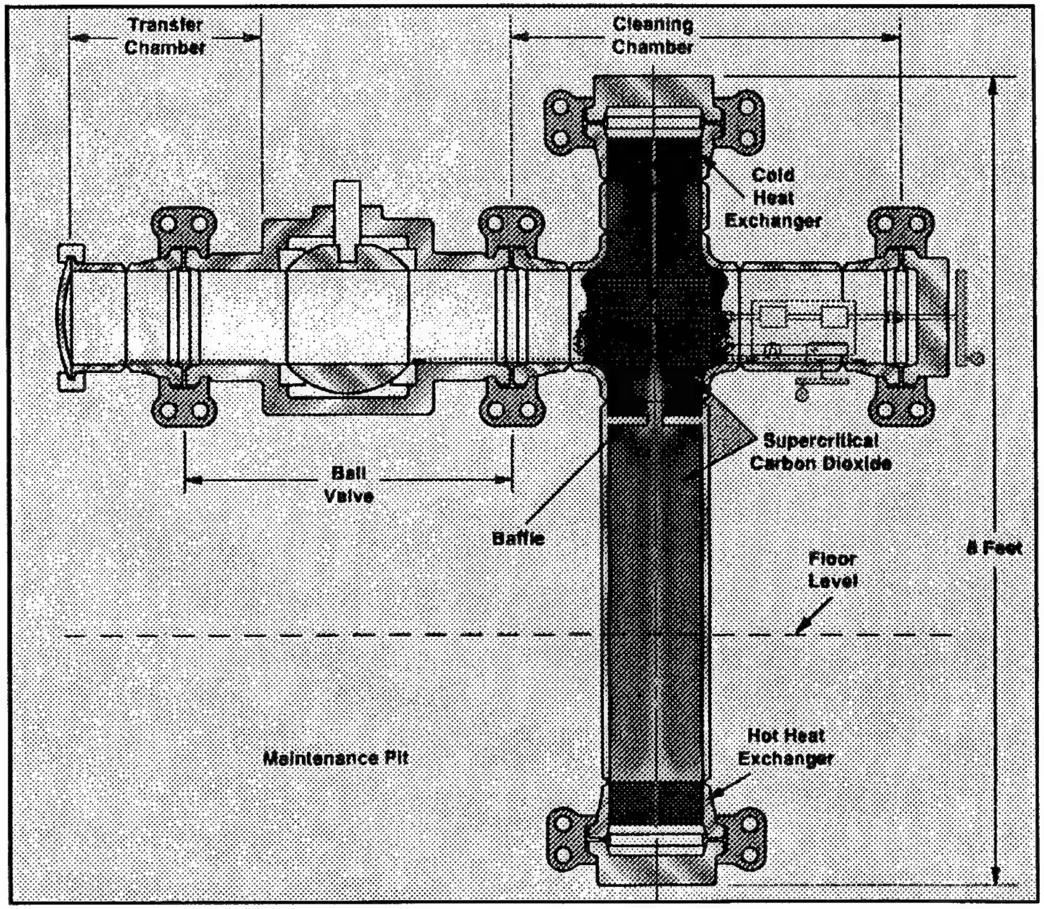


Figure 6. Schematic of Natural Convection Cleaner with Parts Basket in Cleaning Position

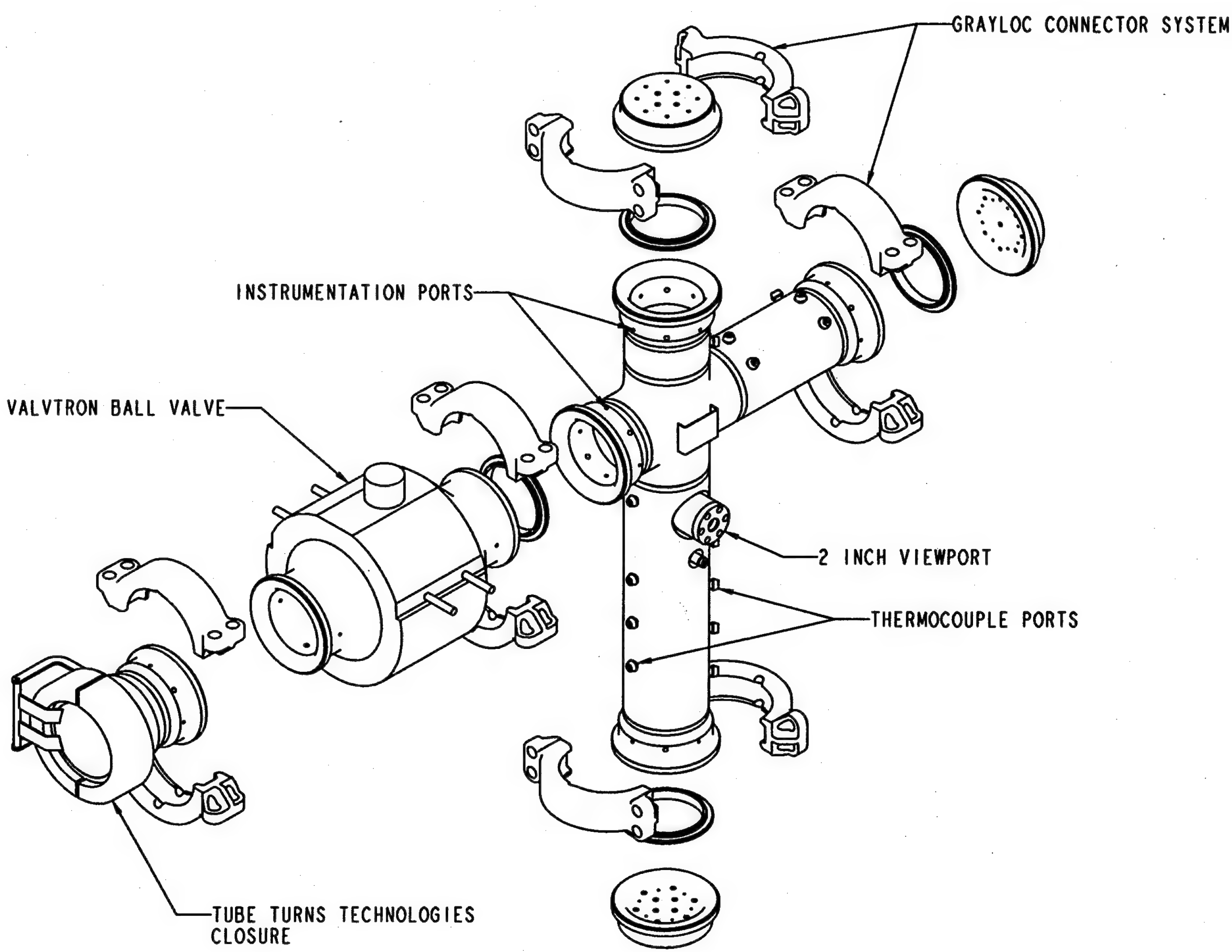


Figure 7. Exploded View of Pre-production Unit

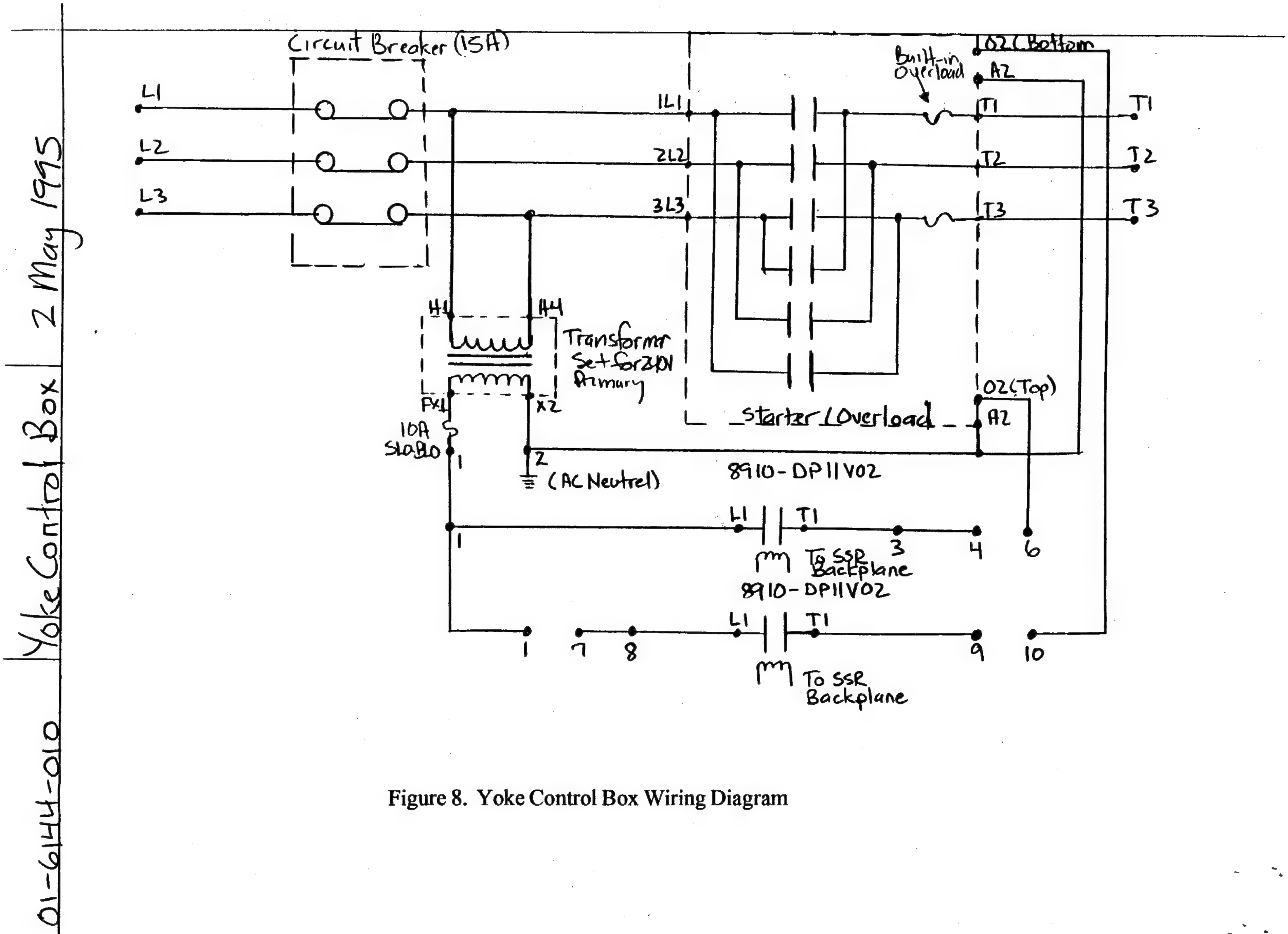


Figure 8. Yoke Control Box Wiring Diagram

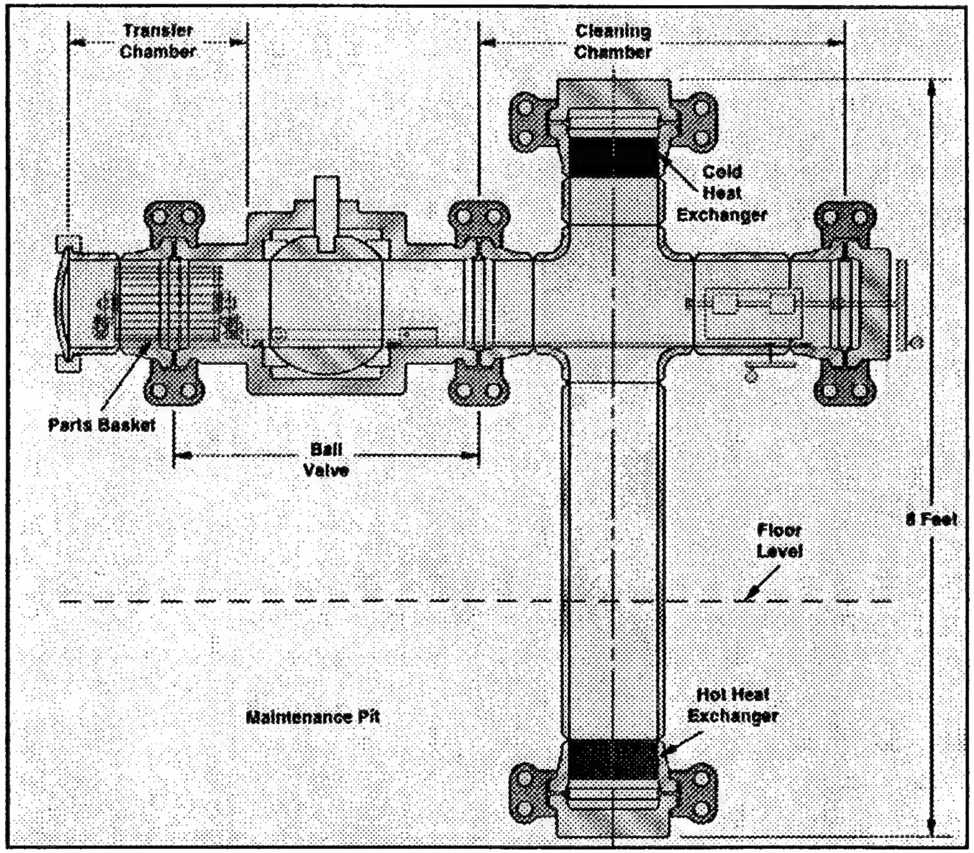


Figure 9. Schematic of Parts Basket Transport Subsystem in Pickup Position

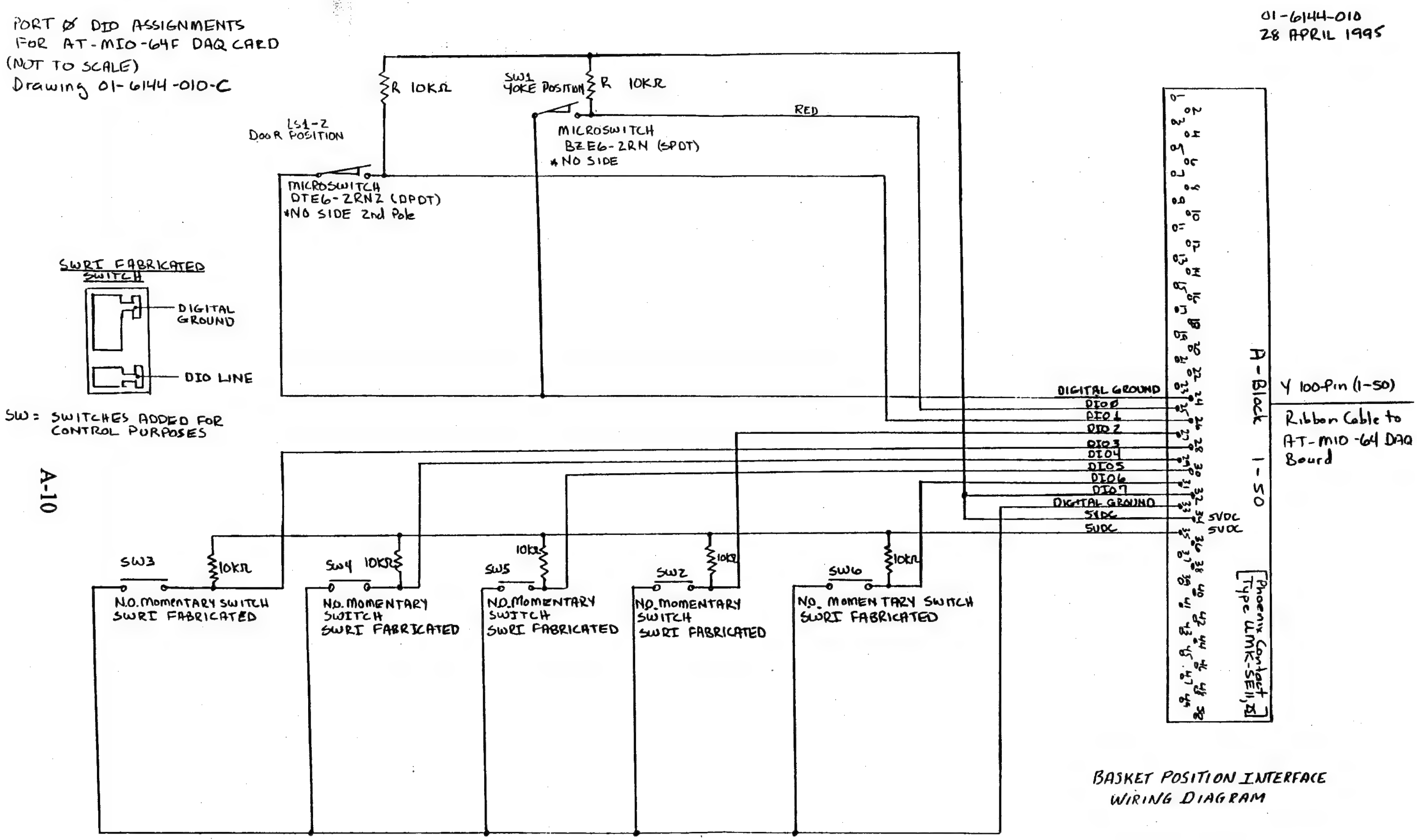


Figure 10. Basket Position Interface Wiring Diagram

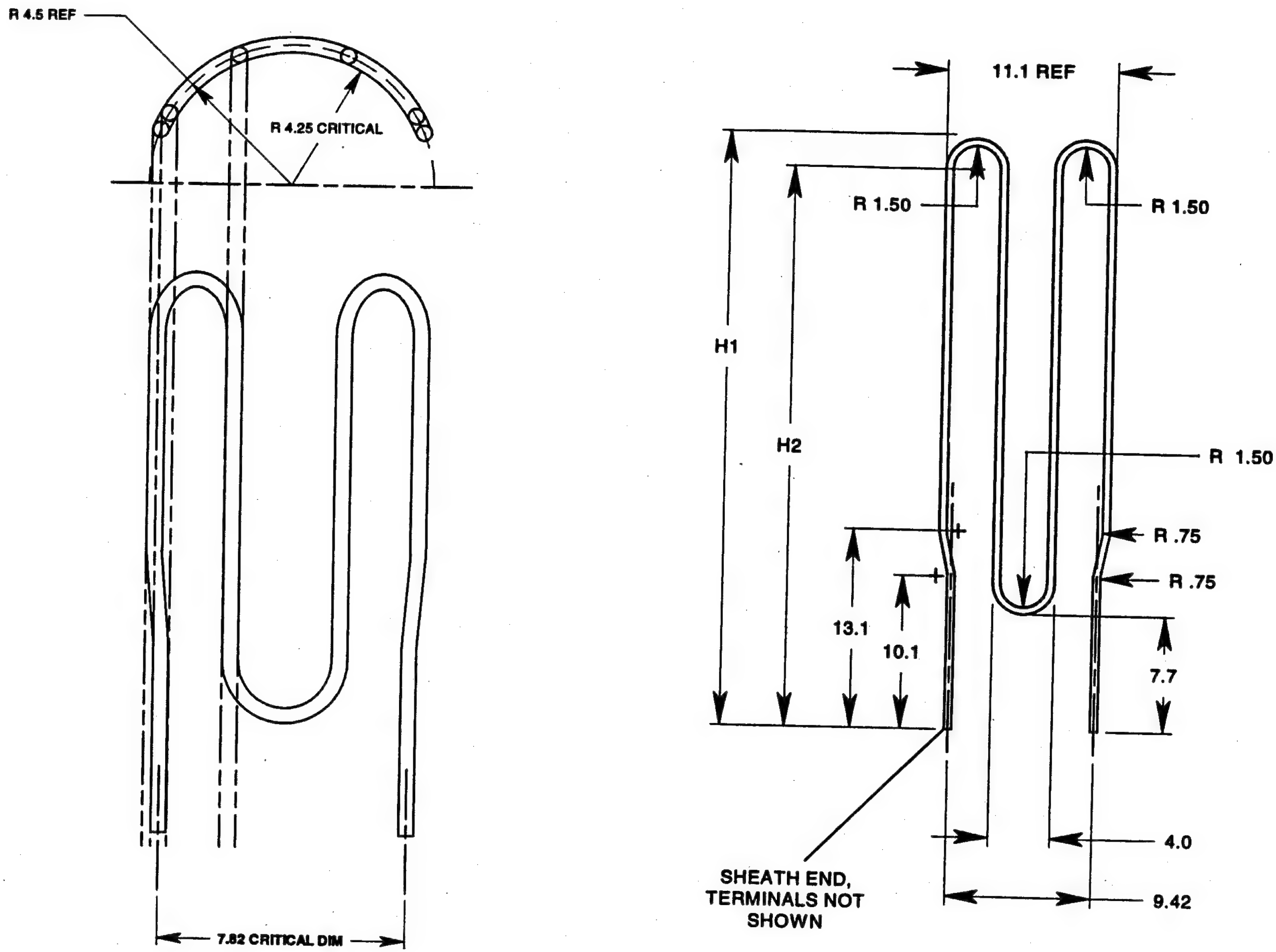


Figure 11. Immersion Heater Design Drawing

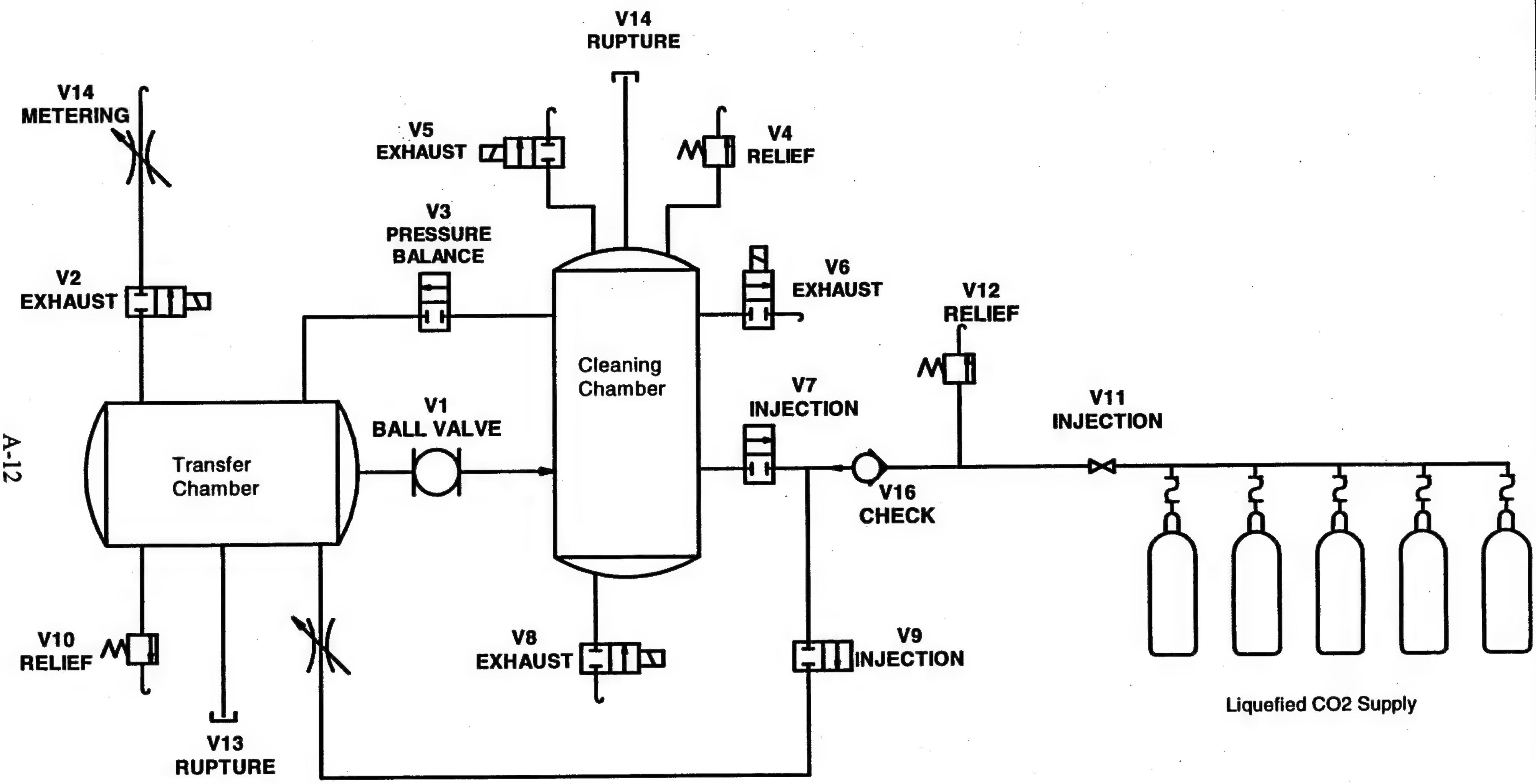


Figure 12. Carbon Dioxide Piping Diagram

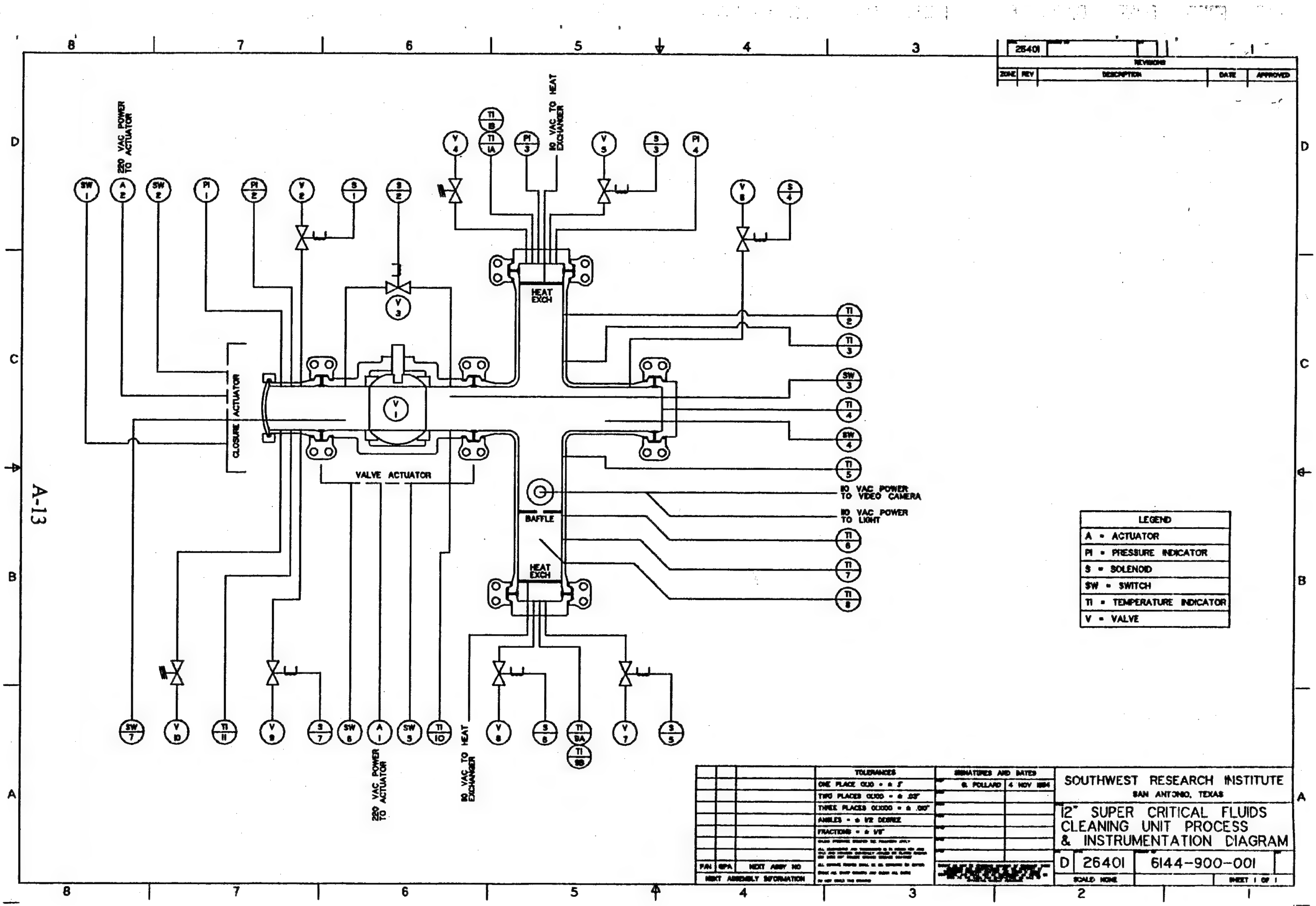
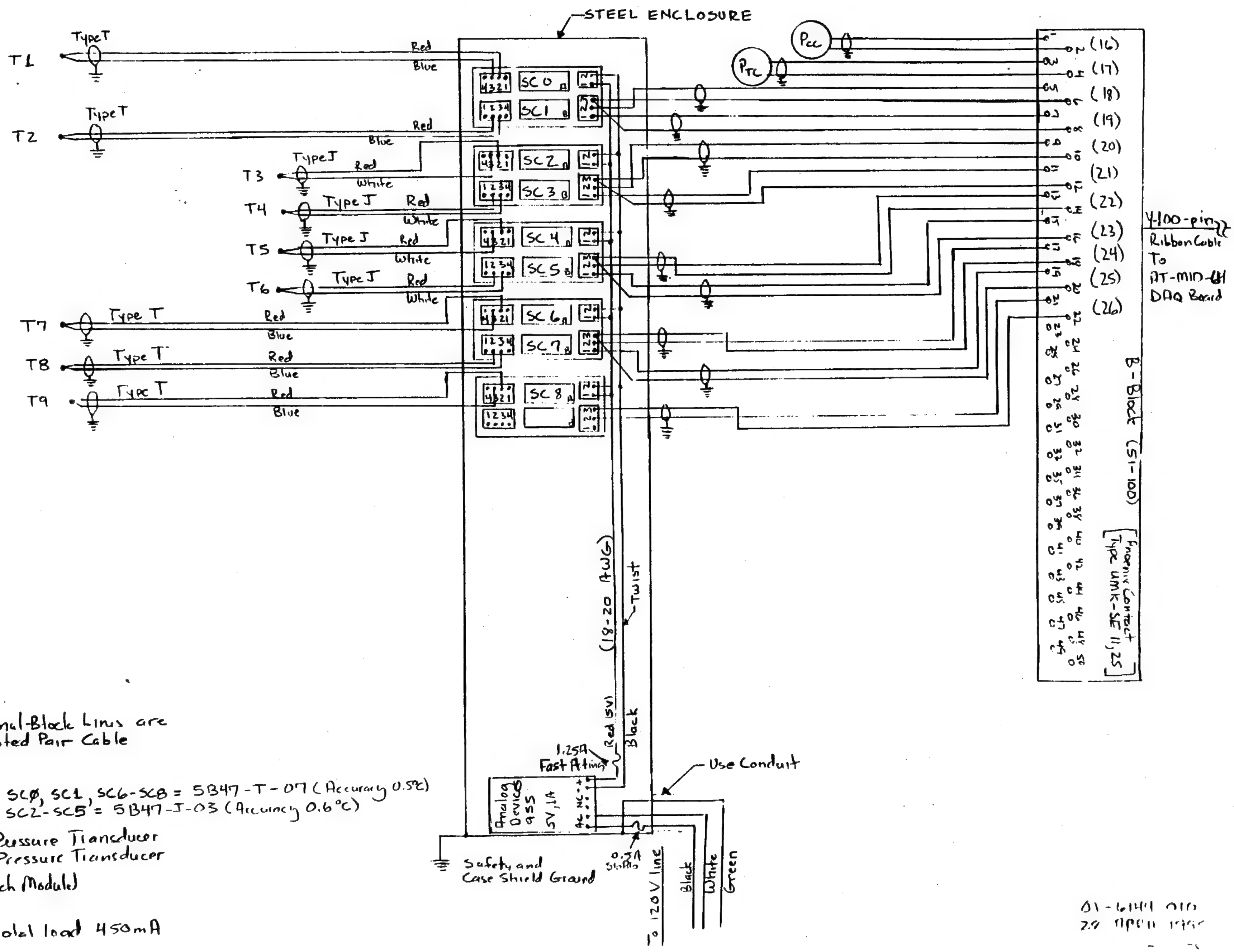


Figure 13. Instrumentation Diagram for Pre-production Cleaner

ANALOG INPUTS FOR
AT-MIO-64F DAQ CARD
(NOT TO SCALE)
Drawing 01-6144-010-B



* All SC-Output-to-Terminal-Block Lines are 22AWG Shielded Twisted Pair Cable

SC = Signal Conditioner SCØ, SC1, SC6-SC8 = 5B47-T-07 (Accuracy 0.5°C)
SC2-SC5 = 5B47-J-03 (Accuracy 0.6°C)
T = Thermocouple
P_{CC} = Cleaning Chamber Pressure Transducer
P_{TC} = Transfer Chamber Pressure Transducer
Power Consumption (Each Module)
150mW (30mA)
Assume 15 installed - total load 450mA

01-6144-010
28 APRIL 1995

Figure 14. Analog Inputs for Data Acquisition Card

BLOCK DIAGRAM OF SYSTEM
(NOT TO SCALE)
1 MAY 1995
DRAWING 01-6144-010-D

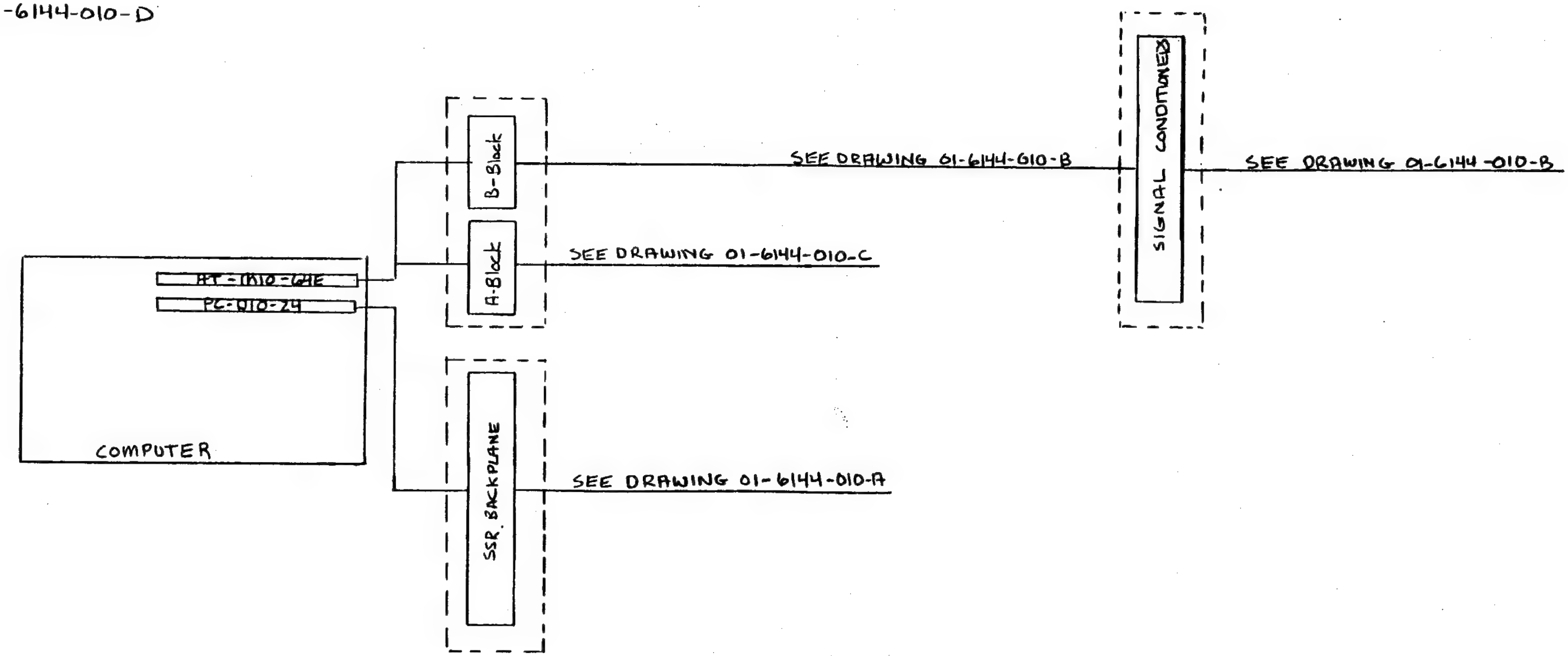


Figure 15. Block Diagram of Computer Control System

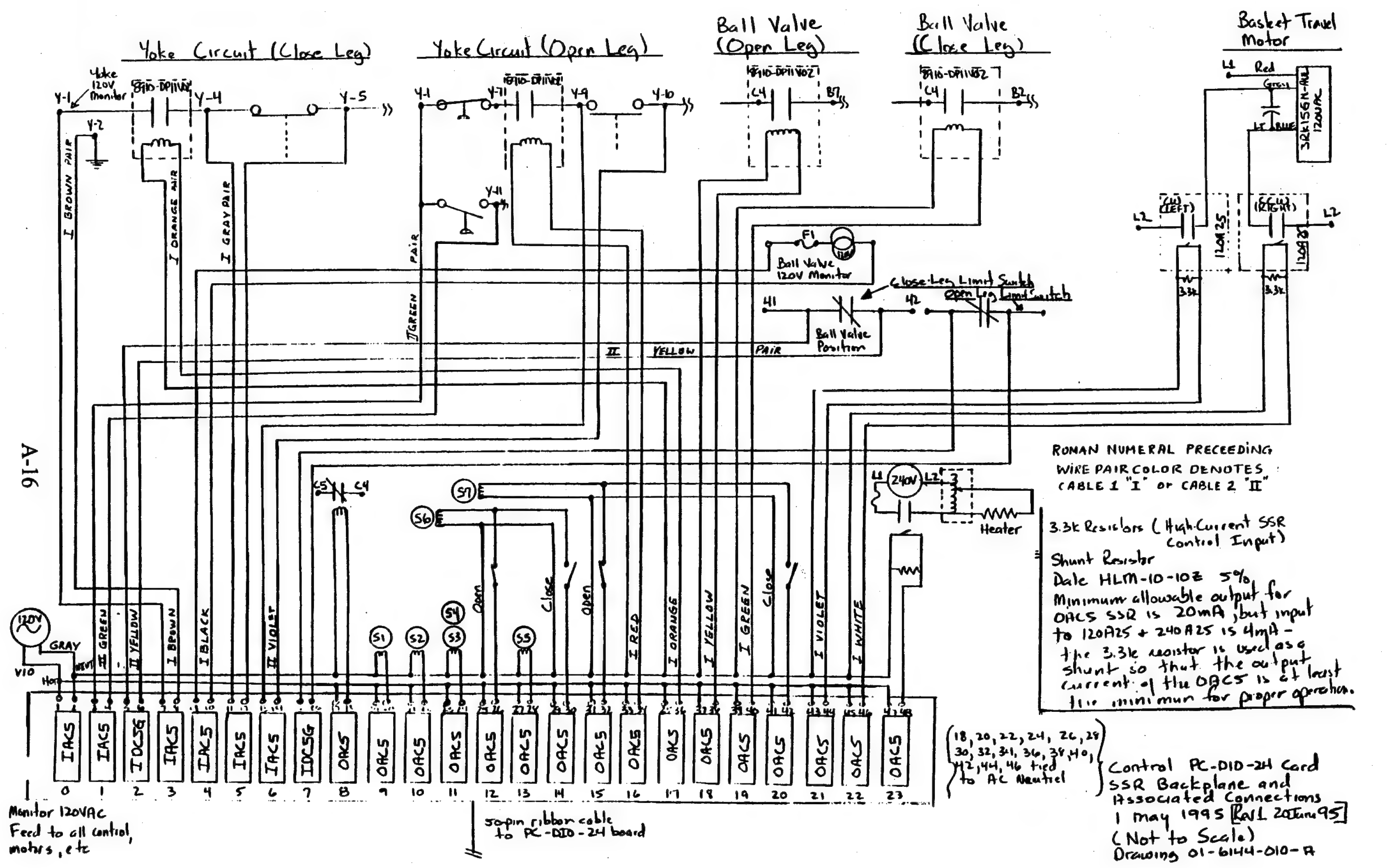


Figure 16. Wiring Connections to Control Card

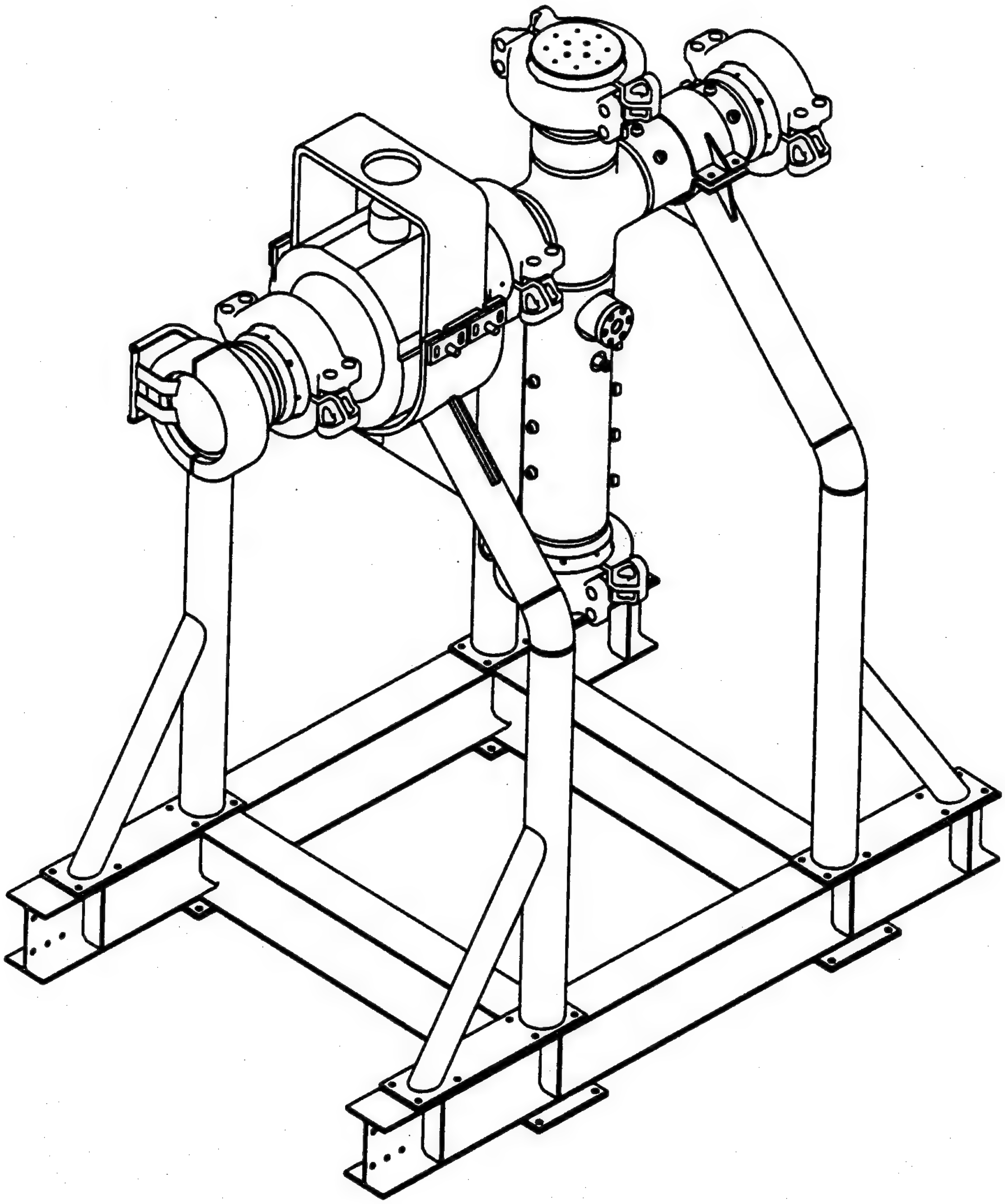


Figure 17. SCF Cleaning Chamber Isometric View

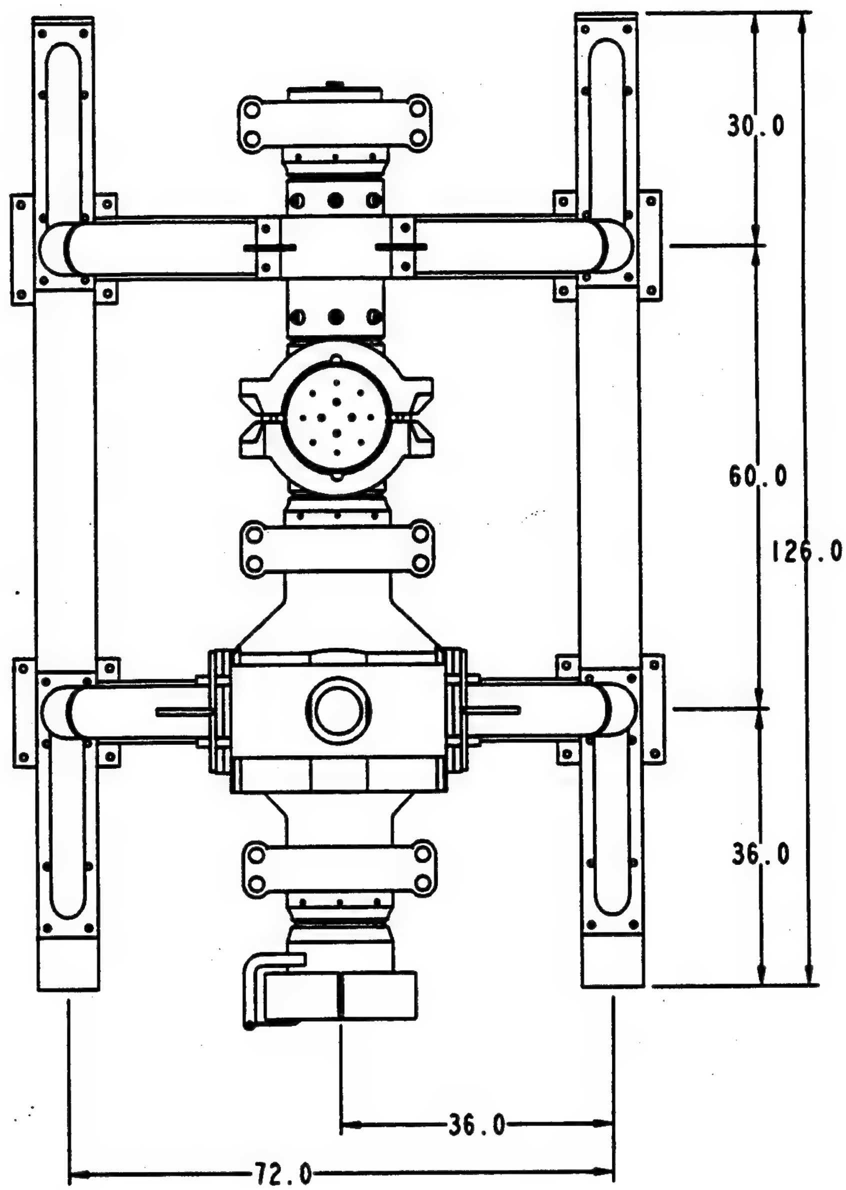


Figure 18. SCF Cleaning Chamber Top View

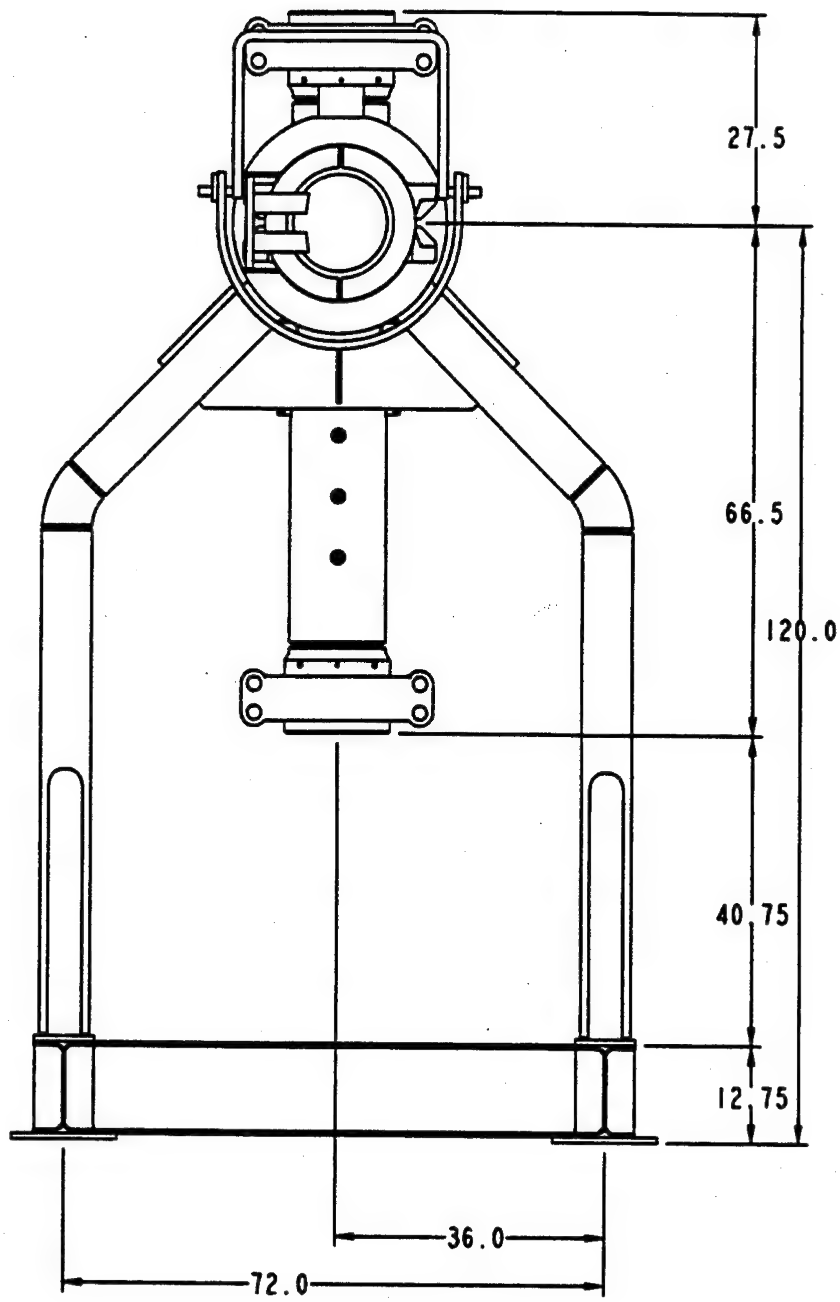


Figure 19. SCF Cleaning Chamber Front View

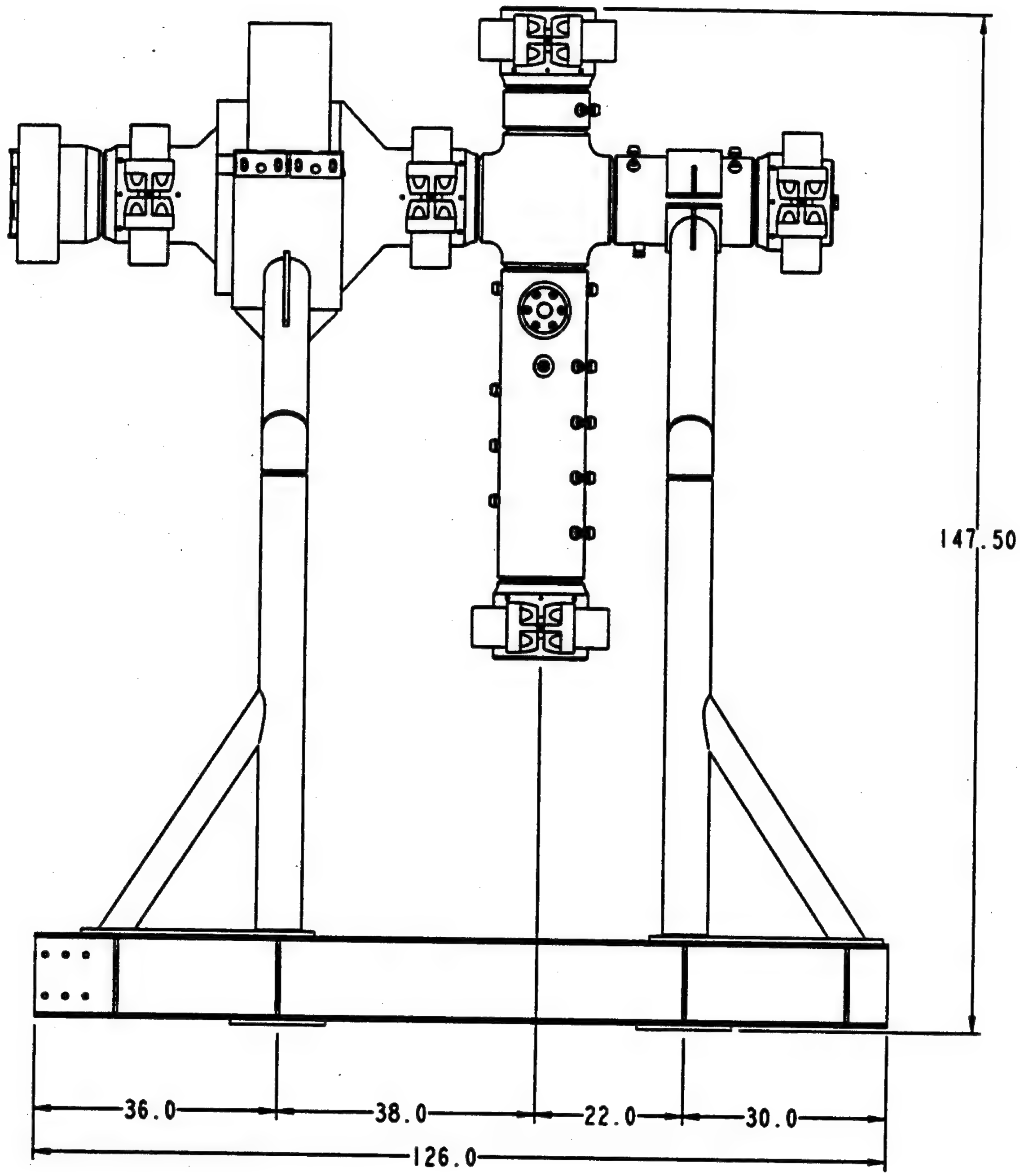


Figure 20. SCF Cleaning Chamber Right Side View

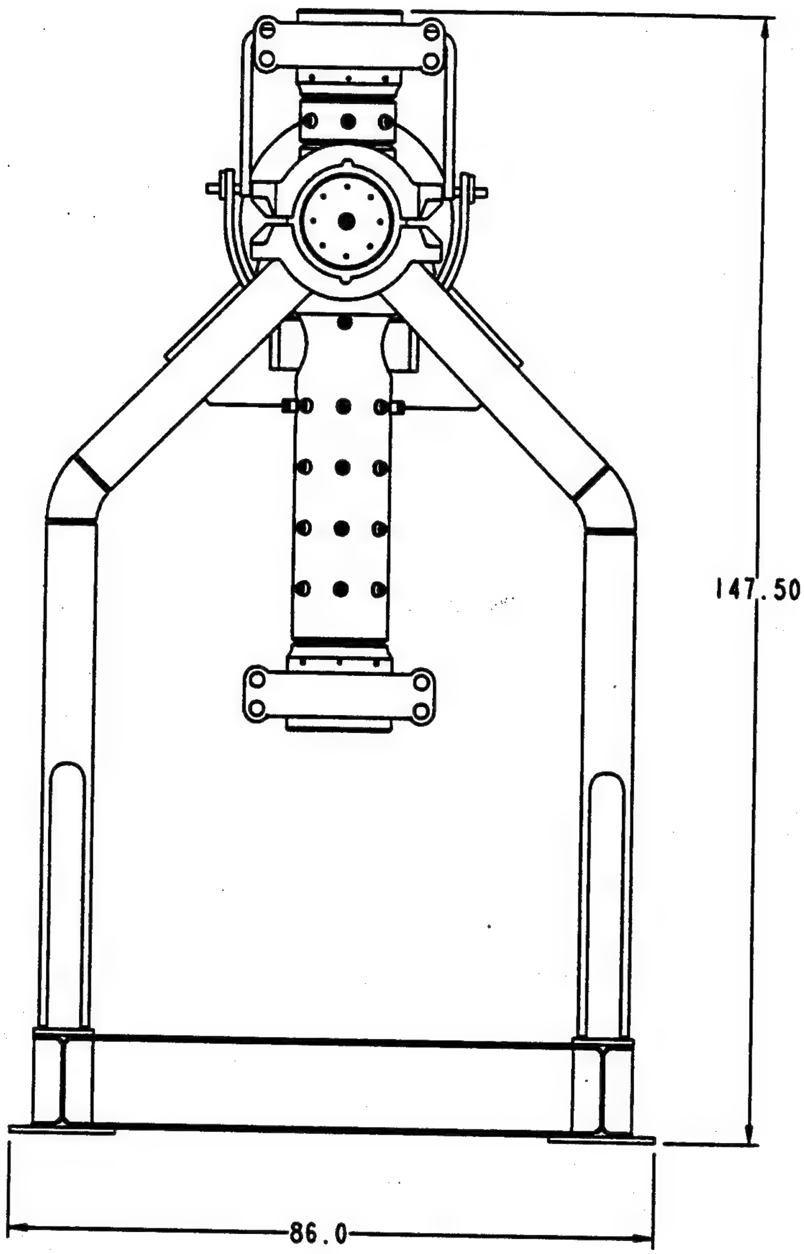


Figure 21. SCF Cleaning Chamber Rear View

APPENDIX B

OPERATING INSTRUCTIONS
for the
NON-OZONE DEPLETING SUPERCRITICAL
CO_2 CLEANING-FLUID SYSTEM

OPERATING INSTRUCTIONS
for the
NON-OZONE_DEPLETING
SUPERCRITICAL CO_2 CLEANING-FLUID SYSTEM

Date Prepared: 21 June 1995
Last Revision: 17 October 1995

Prepared for: United States Air Force

Prepared by: Southwest Research Institute

TABLE OF CONTENTS

INTRODUCTION

This document provides step-by-step instructions for the operation of the non-ozone-depleting supercritical-CO_2 cleaning-fluid system developed by Southwest Research Institute. Instructions include system start-up and shut down, cleaning-chamber filling procedure, performance of automated cleaning cycles, and access to functions on the password-protected *SYSTEM MANAGER CONTROL*, *SYSTEM OPERATING PARAMETERS*, and *SYSTEM DAQ CALIBRATION VALUES* panels. Error codes that could be encountered during system start-up and operation are described in the appendix of this document. Also included in the appendix are descriptions of *SUPERCRITICAL CO_2 CLEANING FLUIDS SYSTEM* (main), *SYSTEM MANAGER CONTROL*, *SYSTEM OPERATING PARAMETERS*, and *SYSTEM DAQ CALIBRATION VALUES* panel controls and their functions.

SYSTEM START-UP

☞ Turn on the system computer and monitor. Wait for computer to boot; the autoexec.bat file contains the necessary code to start Windows at the end of the boot sequence. Windows will automatically load and run the SCF application. After completion of the above sequences, an introductory panel will be displayed on the computer monitor. A "Press Any Key to Continue" instruction is also displayed; however, do not execute this instruction until indicated in the subsequent System Start-Up sequence.

☞ If off, turn on main system circuit breaker (100-amp, 208-volts, 3-phase circuit breaker labeled SCF and located in circuit-breaker box A).

☞ Check the remote/off/local/off switch on the ball-valve actuator. Make sure it is set in the remote position.

☞ If off, turn on secondary circuit breakers (three 20-amp, 208-volts, 3-phase circuit breakers labeled "Closure Control", "Ball-Valve Actuator", and "Lift Table"; one 20-amp, 208-volts, single-phase circuit breaker labeled "Heaters"; and two 20-amp, 110-volt circuit breakers labeled "System Control Voltage" and "Receptacle" (all located in circuit-breaker box B). Note that when the "Receptacle" circuit breaker is turned on, the cooling-system pump will start; this is normal.

☞ If off, turn on the thermoelectric cooling unit and, if necessary, adjust/set the temperature set point.

☞ If off, turn on the closure-control circuit breaker by rotating the externally accessible handle to the on position.

☞ After the all above steps have been performed and confirmed, press any key on the computer keyboard (as instructed on the introductory panel) to continue the program. After a key is pressed, the operator is instructed to wait while the system is initialized. If no errors are encountered, the system's main panel (refer to Figure 1) will be displayed; a description of the main-panel controls and indicators is included in the appendix. If errors are detected during system initialization, the operator is notified and system error codes are displayed on the introductory panel; after recording the error codes, the operator can press any key to terminate the program. The program can be restarted after the error conditions are corrected. A description of error codes is also included in the Appendix.

☞ If the cleaning chamber has not been filled, then refer to the "FILLING CLEANING CHAMBER" section for instructions before proceeding with any cleaning cycles. If the cleaning chamber is already filled, then insure that system is at the proper operating temperature and pressure before initiating a cleaning cycle.

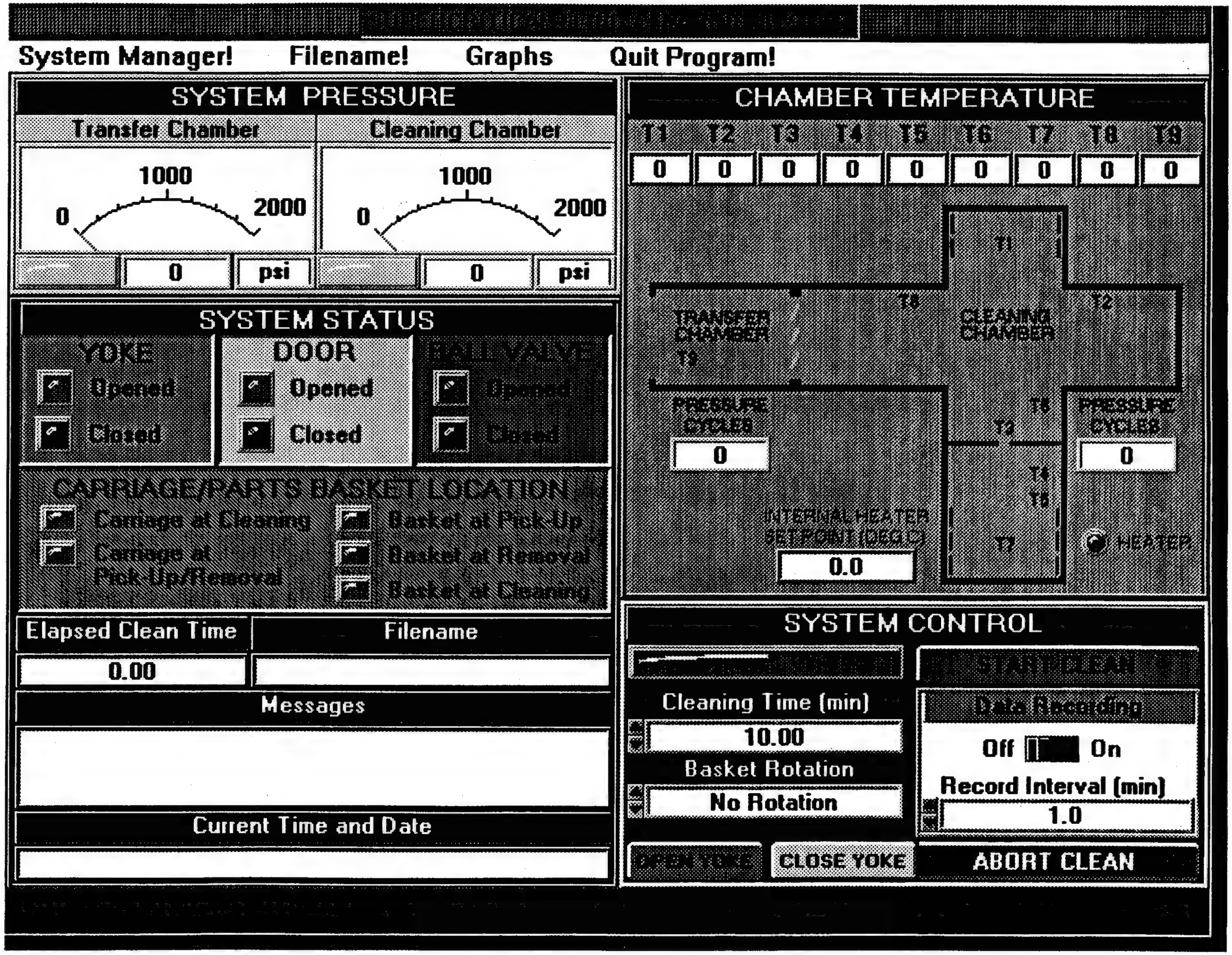


Figure 1. Main (Supercritical CO_2 Cleaning Fluid System) Panel

FILLING CLEANING CHAMBER

The cleaning chamber must be properly filled with CO_2 before any parts cleaning can be performed. Controls needed to fill the cleaning chamber are located on the *SYSTEM MANAGER CONTROL* panel; refer to the "ACCESSING SYSTEM-MANAGER PANEL" section of this manual for instructions on accessing this password protected panel. A description of each *SYSTEM MANAGER CONTROL* functions is included in the appendix; the operator should be familiar with all system-manager controls before performing this procedure.

The fill procedure generally consists of pressurizing the cleaning chamber to approximately 400 psi, then purging until the internal pressure reaches about 100 psi. If other fill-purge pressure limits are used during this stage of the process, make sure that these limits do not create pressure differentials of more than 400 psi; otherwise, a pressure cycle will be recorded for each fill-purge sequence, thus reducing the useful life of the vessel. The fill-purge sequence should repeated two, three or more times to reduce the percentage of ambient gases within the cleaning chamber. After executing the required number of fill-purge cycles, the cleaning chamber is filled with the specified amount (by weight) of liquid CO_2. The number of fill-purge cycles to perform, upper and lower pressure limits to use during the fill-purge cycles, as well as the amount of liquid CO_2 to inject into the cleaning chamber should be determined before starting this procedure.

The following steps will properly fill the cleaning chamber with liquid CO_2.

☞ Make sure that the cleaning-chamber fill valve is in the closed position. If not, close the valve by clicking on the appropriate "CLOSE" button under "Fill Valves" in the Independent Control section of the *SYSTEM MANAGER CONTROL* panel; an indicator above the "OPEN" and "CLOSE" cleaning-chamber fill-valve buttons shows the current position of the fill valve.

☞ Make sure that the ball valve is closed. Note that the cleaning-chamber fill procedure can be performed with the ball valve open; however, the CO_2 that fills the transfer-chamber volume will have to be purged, and therefore wasted, before parts can be placed in the vessel. If you choose to leave the ball valve open, then it will be necessary to close the door and yoke assembly before proceeding. Built-in safety features prevent opening of the cleaning-chamber fill valve if both the ball valve and yoke are open; one or both must be closed before the fill valve can be opened.

☞ Make sure that the liquid CO_2 cylinders are properly connected to corresponding intake ports on the supply manifold and each three-way intake-port valve is closed. Open the shut-off valves on top of each CO_2 cylinder. One at a time, slightly open each three-way intake-port valve to the purge position; this will remove ambient gases from the cylinder-to-manifold supply lines and prevent their introduction into the cleaning chamber. After purging all four cylinder-to-manifold supply lines, turn each three-way intake-port valve to the open position.

FILLING CLEANING CHAMBER (CONT'D.)

A description of the CO_2 supply manifold and associated system can be found in the "CO_2 CYLINDERS, SUPPLY MANIFOLD AND VALVES" section; detailed procedures for changing cylinders, purging lines, and operating manifold three-way intake-port valves are included in the aforementioned section.

☛ Perform the required number of fill-purge cycles; each fill-purge cycle consists of the following steps:

> Open the cleaning-chamber fill valve by clicking on the appropriate "OPEN" button in the Independent Control section of the *SYSTEM MANAGER CONTROL* panel. Note that if both the ball valve and yoke are open or if there is a power failure that prevents checking the status of the ball valve or yoke, the open fill valve operation will be ignored and the operator will be notified via the "System Messages" indicator; otherwise, the fill valve will open and a corresponding message will be posted in the "System Messages" indicator.
>
> Allow the cleaning chamber to reach the specified fill pressure, then close the cleaning-chamber fill valve using the appropriate "CLOSE" button on the *SYSTEM MANAGER CONTROL* panel.
>
> Open both the cleaning-chamber purge valve and contaminant-removal valve by placing the appropriate switches in the "Open" positions.
>
> Allow the cleaning-chamber to vent until the pressure reaches the specified purge pressure, then close the cleaning-chamber purge valve and contaminant-removal valve.

☛ After completing the required number of fill-purge cycles, either record the current weight of the CO_2 cylinders or zero the scale.

☛ Open the cleaning-chamber fill valve as well as the cleaning-chamber purge valve. Do not open the contaminant-removal valve.

☛ After the required quantity (by weight) of liquid CO_2 has been siphoned into the cleaning chamber, close both the cleaning-chamber fill and purge valves. If it is necessary to change CO_2 cylinders during the fill procedure, then simply close both the cleaning-chamber fill and purge valves via system-manager controls, replace the cylinders as described in the "CO_2 CYLINDERS, SUPPLY MANIFOLD AND VALVES" section of this manual. After new CO_2 cylinders are connected, be sure to purge each cylinder-to-manifold supply line before placing the three-way intake-port valves in the open position. Next, record the weight of the CO_2 cylinders (or zero the scale), then open both the cleaning-chamber fill and purge valves. The cylinders can be changed as often as needed; however, be sure to accurately total all siphoned liquid CO_2.

AUTOMATIC CLEANING CYCLE

Before the automatic cleaning cycle can be used, the cleaning chamber must be filled with CO_2 and pressure and temperature should be at the required operating points. These parameters are NOT checked by the control software when an automatic cleaning cycle is initiated; therefore, it is the operator's responsibility to insure that the system is at the correct pressure and temperature. All system settings have default values; if default values are acceptable, then no changes need to be made to the system controls. A description of main-panel controls and indicators as well as menu-bar items is included in the appendix.

It is also important that the cylinders contain enough liquid CO_2 to fill the transfer chamber during the automatic cleaning cycle. There is no provision for closing the fill valve if cylinders become empty before the required amount of liquid CO_2 has been introduced into the transfer chamber. If it becomes absolutely necessary to change the cylinders during this part of the automatic cleaning cycle, then close all three-way intake-port valves on the supply manifold and the metering valve that is in-line with the transfer-chamber purge valve, replace the cylinders, purge the cylinder-to-manifold supply lines, and open the three-way intake-port valves and the transfer-chamber metering valve.

☞ Set the desired cleaning time by entering this time into the "Cleaning Time" control located in the "SYSTEM CONTROL" block on the main panel. Time is entered in minutes.

☞ Select desired basket rotation (No Rotation, 90° Degrees, and Continuous Rotation) from the "Basket Rotation" control in the "SYSTEM CONTROL" block on the main panel.

☞ If data recording is desired, then set the "Data Recording" switch (located in the "SYSTEM CONTROL" block on the main panel) to the "On" position.

☞ If data-recording has been selected ("Data Recording" switch on), then the "Record Interval" control (located in the "SYSTEM CONTROL" block on the main panel) will be enabled. If an interval other than the current default is desired, then enter the new interval into the control; recording interval is entered in minutes.

☞ If desired, change the data-recording filename; otherwise, the new data will be appended to the default data-recording file (scfdef.dat). The current filename is displayed in the "Filename" indicator in the "SYSTEM STATUS" block. A new filename can be entered by selecting "Filename!" menu-bar item from the horizontal menu bar. When selected, a small pseudo panel is displayed over the "SYSTEM CONTROL" block of the main panel (refer to Figure 2). Enter the filename in the appropriate control, then press the "OK" button. A "CANCEL" button is also provided so that the filename-entry operation can be discontinued without changing the filename.

☞ Load the parts basket with the parts to be cleaned. Evenly distribute the parts by weight to minimize load on the motor.

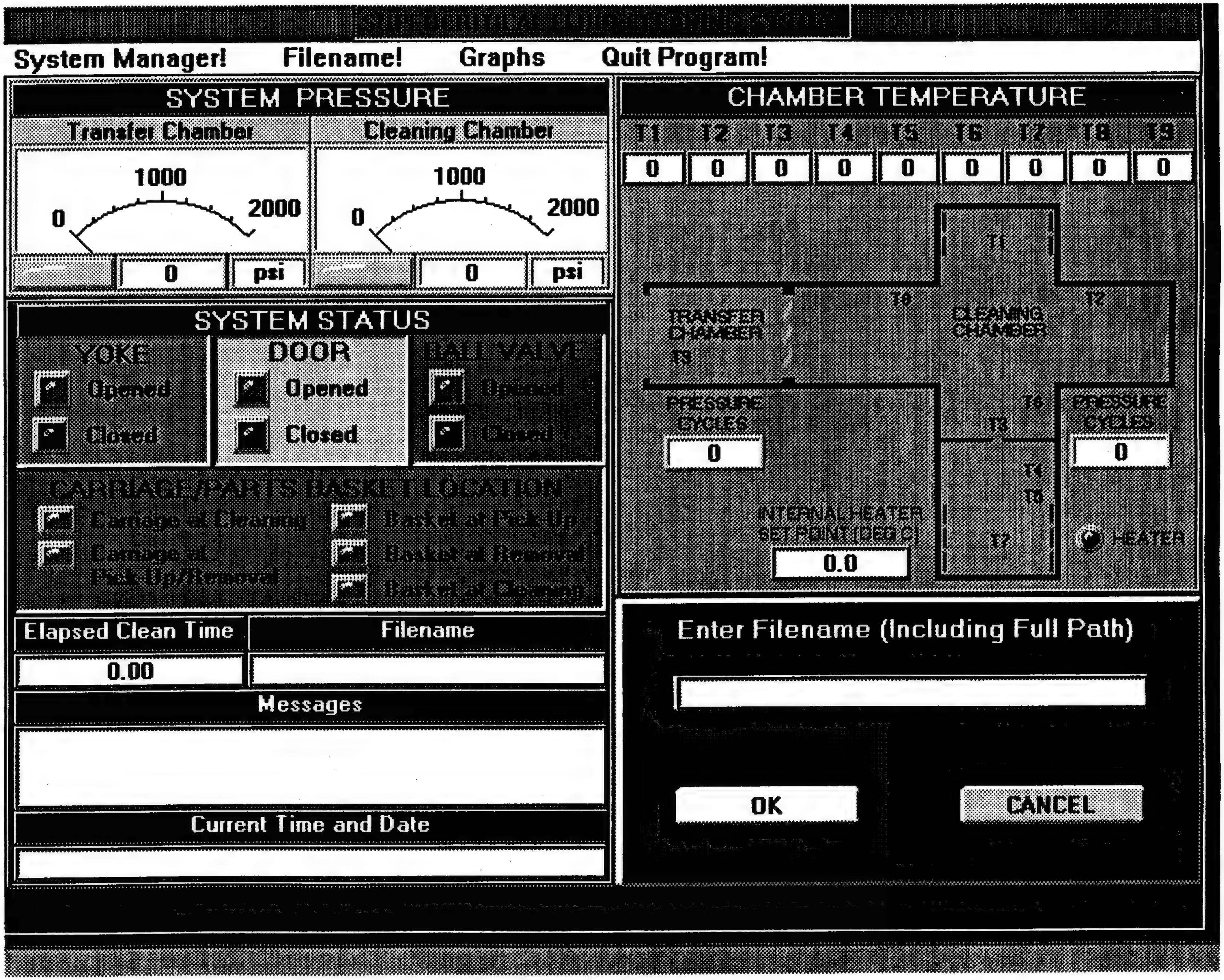


Figure 2. Main Panel With Filename Entry Block Displayed

AUTOMATIC CLEANING CYCLE (CONT'D.)

☞ Open the door to the SCF unit and place the parts basket into the chamber. Push the parts basket into the chamber until it contacts the closed ball valve. This insures that the basket is in the correct location for pickup. The "Basket at Pick-Up" LED indicator, located in the "SYSTEM STATUS" block under the "CARRIAGE/PARTS BASKET POSITION" section, will be lit when the basket is properly positioned.

☞ Close the door to the SCF unit. The "Closed" door position LED indicator, located in the "SYSTEM STATUS" block under the "DOOR" section, will light when the door is properly closed; the cross-sectional diagram will also indicate door position.

☞ Using the mouse, point and click on the "START CLEAN" button in the "SYSTEM CONTROL" block on the main panel. When the cleaning cycle is initiated, the system verifies several parameters including door in closed position; parts basket in pick-up position; carriage in cleaning-chamber position; and voltage to closure unit (yoke), ball-valve actuator, limit switches, and other data-acquisition and control functions. Error messages are displayed in the "Message" indicator in the "SYSTEM STATUS" block on the main panel and the cleaning cycle is prevented from starting if the states of any of the aforementioned parameters are incorrect. A description of error codes is included in the appendix of this document.

☞ After executing the required number of fill-purge sequences to reduce the percentage of ambient gases in the transfer chamber, a "LIQUID CO_2 FILL" button will be displayed over the "START CLEAN" button in the "SYSTEM CONTROL" block. Before continuing with the automatic clean cycle, the operator needs to record the weight of the CO_2 cylinders or zero the scale so the amount of liquid CO_2 siphoned into the transfer chamber can be controlled. Press the "LIQUID CO_2 FILL" button after the weight has been recorded.

☞ When the liquid CO_2 fill has started, a "CONTINUE CLEAN" button will be displayed over the "START CLEAN" button in the "SYSTEM CONTROL" block. After the proper amount of liquid CO_2 has been siphoned into the transfer chamber, press the "CONTINUE CLEAN" button to close the fill valve and continue the automatic cleaning cycle. If the CO_2 cylinders must be changed during this process, then proceed as described in the second paragraph of this section. **DO NOT** press the "CONTINUE CLEAN" button to change cylinders as this will cause the transfer-chamber fill valve to close and the automatic cleaning cycle to proceed with the next step regardless of the quantity of liquid CO_2 in the transfer chamber.

Note that during the cleaning cycle, all "SYSTEM CONTROL" block controls, except the "ABORT CLEAN" button are disabled; all menu-bar items, except for "Graphs" are also disabled. Some temperature indicators are disabled during performance of many of the cleaning-cycle support functions (e.g., moving carriage, closing ball valve and yoke, purging and filling transfer chamber, etc.); the "Graphs" menu-bar item is also disabled during execution of support functions. After parts have been properly positioned in the cleaning chamber, the "Elapsed Clean

AUTOMATIC CLEANING CYCLE (CONT'D.)

Time" indicator (located in the "SYSTEM STATUS" block) will begin to increment; elapsed cleaning time is shown in minutes. During actual cleaning, the operator can display graphs showing histories of system temperatures and pressures. Graphs are accessed by clicking on the "Graphs" menu-bar item. A pull down menu will present the user with two additional choices: Temperature (T1/T7,T2,T8 and T9) and Temperature (T3-T6). The operator can select the desired graphs by clicking on the appropriate pull-down menu item. If the cleaning cycle ends before the operator returns to the main panel, then the program will automatically remove the displayed graphs and redisplay the main panel. The graphs can be examined after remaining cleaning-cycle support functions have been executed.

ACCESSING SYSTEM MANAGER CONTROL PANEL

The *SYSTEM MANAGER CONTROL* panel is password protected. Only those individuals with the correct password are allowed access to this panel. The "System Manager" menu-bar item (main panel) is enabled when no support functions (e.g., opening or closing yoke, selecting a filename, etc.) are being performed and when a cleaning cycle is not underway. A description of the controls and indicators on the *SYSTEM MANAGER CONTROL* panel is included in the appendix at the end of this document.

☞ Click on the "System Manager" menu-bar item of the horizontal menu bar. A pseudo password panel will be display over the "SYSTEM CONTROL" block of the main panel (refer to Figure 3). The password panel contains a control for password entry as well as two buttons, one labeled "OK" and the other labeled "CANCEL". The "CANCEL" button can be used to terminate the password-entry procedure at any time while the password-entry panel is displayed.

☞ Enter the password into the appropriate control. The password that is entered will not be visible on the screen. Note that the password is case sensitive.

☞ Click on the "OK" button. If the correct password was entered, the *SYSTEM MANAGER CONTROL* panel will be displayed; otherwise, a message will be displayed in the message window, located in the "SYSTEM STATUS" block of the main panel, informing the operator of an incorrect password.

To exit the *SYSTEM MANAGER CONTROL* panel, click on the "RETURN TO MAIN PANEL" button.

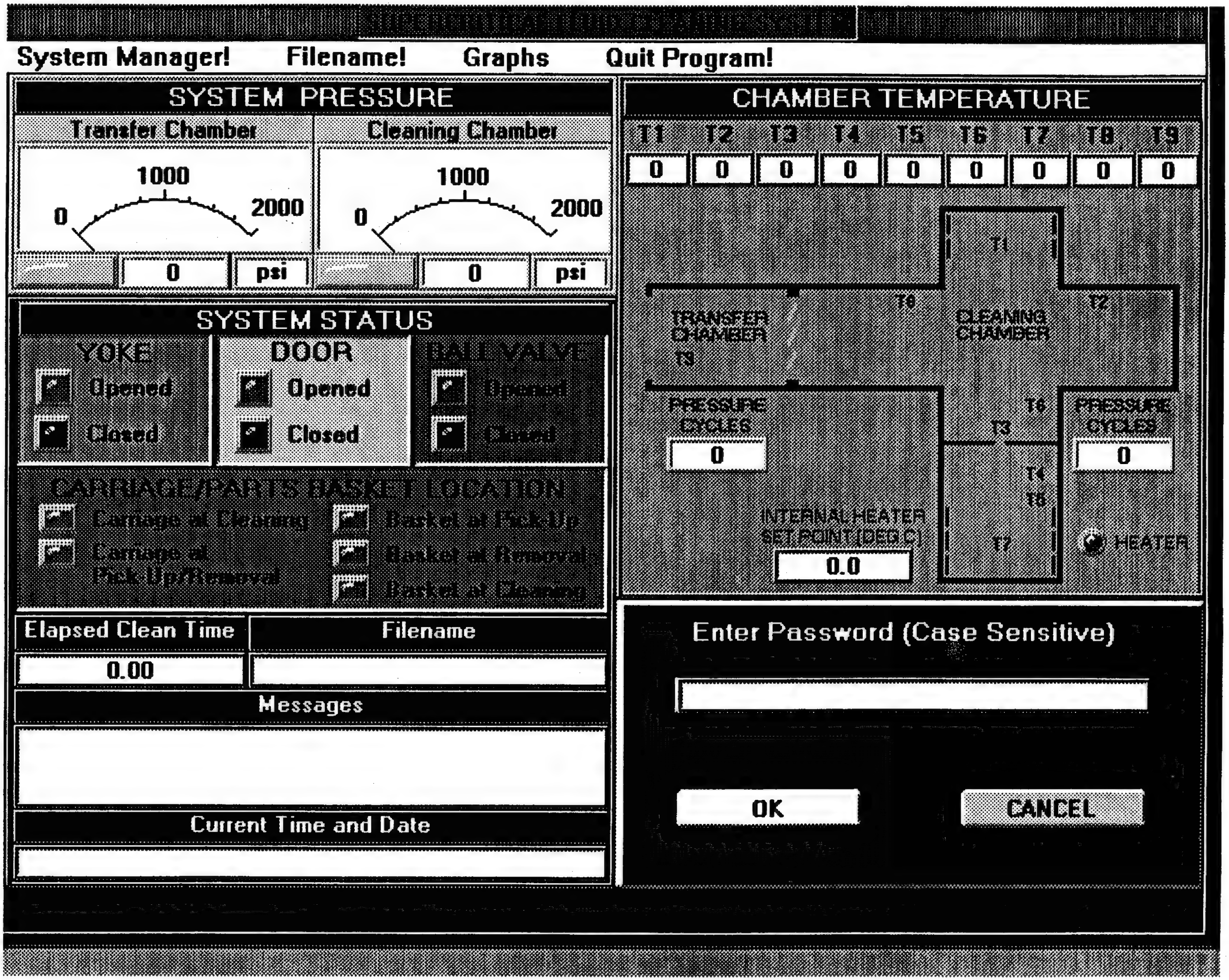


Figure 3. Main Panel With Password Entry Block to System Manager Control Panel Displayed

ACCESSING SYSTEM OPERATING PARAMETERS PANEL

The *SYSTEM OPERATING PARAMETERS* panel can only be accessed from the *SYSTEM MANAGER CONTROL* panel; therefore, it can only be accessed by those individuals with the correct password. To display the *SYSTEM OPERATING PARAMETERS* panel, simply click on the "SET SYSTEM PARAMETERS" button located in the lower right corner of the *SYSTEM MANAGER CONTROL* panel. To exit the *SYSTEM OPERATING PARAMETERS* panel click on the "RETURN TO SYSTEM MANAGER" button. A description of the controls on the *SYSTEM OPERATING PARAMETERS* panel is included in the appendix at the end of this document.

ACCESSING SYSTEM DAQ CALIBRATION VALUES PANEL

The *SYSTEM DAQ CALIBRATION VALUES* panel can only be accessed from the *SYSTEM MANAGER CONTROL* panel; therefore, it can only be accessed by those individuals with the correct password. To display the *SYSTEM DAQ CALIBRATION VALUES* panel, simply click on the "SET CALIBRATION VALUES" button located in the lower right corner of the *SYSTEM MANAGER CONTROL* panel. To exit the *SYSTEM DAQ CALIBRATION VALUES* panel click on the "RETURN TO SYSTEM MANAGER" button. A description of the controls on the *SYSTEM DAQ CALIBRATION VALUES* panel is included in the appendix at the end of this document.

SYSTEM SHUT DOWN

☞ Click on the "Quit Program" menu-bar item from the horizontal menu bar; the program will be exited and the Windows desktop will be displayed. Note that the program cannot be terminated while a cleaning cycle is underway or if the yoke is in the process of being opened or closed.

☞ Turn off the thermoelectric cooling unit.

☞ Turn off secondary circuit breakers (three 20-amp, 208-volts, 3-phase circuit breakers labeled "Enclosure Control", "Ball-Valve Actuator", and "Lift Table"; one 20-amp, 208-volts, single-phase circuit breaker labeled "Heaters"; and two 20-amp, 110-volt circuit breakers labeled "System Control Voltage" and "Receptacle" (all located in circuit-breaker box B). Note that the cooling-system pump will shut down when the "Receptacle" circuit breaker is turned off.

☞ Although not absolutely necessary, the closure-control circuit breaker can be turned off by rotating the externally accessible handle to the off position.

☞ Although not absolutely necessary, turn off main system circuit breaker (100-amp, 208-volts, 3-phase circuit breaker labeled SCF and located in circuit-breaker box A).

☞ Exit Windows, wait for the DOS prompt, and then turn off the system computer and monitor.

APPENDIX

ERROR CODES

E1 No 5VDC power to critical control components. Check 5VDC output from AT-MIO-64E board at pins 34 or 35; if absent, check fuse on AT-MIO-64E board.

E2 No 120VAC power to critical control components including chamber solenoid valves, basket travel and rotation motors, and signal-conditioner and pressure-transducer power supplies. Check circuit breaker.

E3 No 120VAC power to yoke control circuitry. Check three-phase circuit breaker, closure-control circuit breaker, and fuse on step-down transformer.

E4 No 120VAC power to ball-valve actuator control circuitry. Check three phase circuit breaker and fuses on step-down transformer.

E5 The closure door is opened and must be closed before the cleaning cycle can be started.

E6 The parts basket has not been placed in the proper pickup position in the transfer chamber.

E7 The carriage is not in the proper position in the cleaning chamber. Use the appropriate system-manager panel function to move the carriage to the proper position.

MAIN PANEL FUNCTIONS

The main (*SUPERCRITICAL CO_2 CLEANING FLUIDS SYSTEM*) panel provides the operator with information on the system status as well as control of high-level-functions (e.g., automatic cleaning cycle). It consists of four main blocks which include "SYSTEM PRESSURE", "SYSTEM STATUS", "CHAMBER TEMPERATURE", and "SYSTEM CONTROL" (Refer to Figure 1).

The "SYSTEM PRESSURE" block contains both analog and digital indicators for transfer- and cleaning-chamber pressures. Within the "SYSTEM STATUS" block are LED indicators the provide the operator with yoke, door, ball valve, basket and carriage positions. Below the LED's are the "Elapsed Clean Time" and "Filename" indicators; these indicators show the elapsed cleaning time in minutes and the currently selected data-storage filename, respectively. A "Message" indicator provides a means of informing the operator of system progress and faults. Current time and date is posted in the "Current Time and Date" indicator.

The "CHAMBER TEMPERATURE" block contains digital indicators for chamber temperatures. The cross-sectional view of the SCF unit shows the approximate location of the thermocouples from which the temperatures are derived.

Finally, the "SYSTEM CONTROL" block provides the operator with the necessary controls to perform an automatic cleaning cycle. These controls include:

"START CLEAN" Button: Allows the operator to initiate an automatic cleaning cycle. Before the cleaning cycle is started, several system parameters are checked for correctness. For example, the basket must be in the pick-up position, the carriage must be in the cleaning-chamber position, the ball valve and door must be closed, and power must be available to all circuitry. If any of the above conditions are incorrect, a message is posted for the operator and the request is ignored.

"LIQUID CO_2 FILL" Button: Allows the operator to record the current weight of the CO_2 cylinders prior to filling the transfer chamber. The automatic cleaning cycle waits for the operator to press this button before continuing with subsequent steps. This button is only visible after completion of the transfer-chamber fill-purge cycles and remains visible only until activated by the operator.

MAIN PANEL FUNCTIONS (CONT'D.)

"CONTINUE CLEAN" Button:	Allows the operator to halt the liquid CO_2 fill sequence of the automatic cleaning cycle when the required quantity (by weight) of liquid CO_2 has been siphoned into the transfer chamber. The automatic cleaning cycle continues with the liquid CO_2 fill until the operator presses this button. This button is only visible during the liquid CO_2 fill portion of the automatic cleaning cycle.
"Cleaning Time" Set-Point Control:	This control provides a means for the operator to vary the total time a part is cleaned during the automatic cleaning cycle; time is entered in minutes.
"Basket Rotation" Set-Point Control:	This control allows the operator to select the basket-rotation scheme. Choices include "No Rotation", 90 Degree, and"Continuous Rotation". If "90 Degree" rotation is selected then, the rotation interval is determined by dividing the total cleaning time by four so that the basket will rotate 360 degrees during the course of the cleaning cycle.
"OPEN YOKE" Button:	Allows the operator to open the yoke provided the difference in pressure between the transfer chamber and atmosphere is within an acceptable (safe) range.
"CLOSE YOKE" Button:	Allows the operator to close the yoke provided the door is in the closed position.
"Data Recording On/Off" Slide Switch	This switch allows the operator to select whether or not temperature, pressure, time and date data is stored to a permanent file during a cleaning cycle. The filename of the currently selected file is displayed in the "Filename" indicator under the "SYSTEM STATUS" block.
"Record Interval" Set-Point Control	Allows the operator to specify the time interval (in minutes) between storing system data to permanent file. This control is disabled if the "Data Recording On/Off" slide switch is set to the off position.

MAIN PANEL FUNCTIONS (CONT'D.)

"ABORT CLEAN" Button — This button, which is enabled only during a cleaning cycle, allows to operator to abort the cleaning cycle for whatever reasons. The abort is not abrupt, but instead, the process is reversed so that parts can be removed and the system is ready for another cleaning cycle.

With the exception of the "ABORT CLEAN" button, controls in the main-panel "SYSTEM CONTROL" block are disabled during a cleaning cycle. All controls are disabled when an open or close yoke sequence is in progress. In addition, during some most cleaning-cycle subfunctions, many of the temperature indicators are not updated; these indicators are blacked out so it is obvious to the operator that they are not active.

The main panel also has a horizontal menu bar so that the operator can access additional system functions.

"System Manager!" — This menu item allows the operator to enter a password for access to the functions on the system manager panel. The password-entry pseudo panel (refer to Figure ??) is displayed over the "SYSTEM CONTROL" block and contains a control for password entry as well as "OK" and "CANCEL" buttons; the "OK" button is selected after the password has been entered and the "CANCEL" button is used to terminate the password-entry process.

"Filename!" — This menu item allows the operator to enter a new filename for data storage. The filename-entry pseudo panel (refer to Figure ??) is displayed over the "SYSTEM CONTROL" block and contains a control for filename entry as well as "OK" and "CANCEL" buttons; the "OK" button is selected after the filename has been entered and the "CANCEL" button is used to terminate the filename-entry process and retain the current filename.

"Graphs" — This menu item allows the operator to view one of three panels containing stripcharts showing system temperatures and pressures. There is a pull down menu with three selections including "Pressure", "Temperature (T1/T7,T2,T8,T9)", and "Temperature (T3-T6). This menu item is available during the actual cleaning portion of the cleaning cycle so that system parameters can be reviewed.

"Quit Program!" — This allows the operator to terminate the SCF program and return to the Windows environment. It is available only when no cleaning cycle or other main-panel activity is in progress.

SYSTEM MANAGER CONTROL PANEL FUNCTIONS

The *SYSTEM MANAGER CONTROL* panel, which is password protected, provides the operator with low-level control over all system functions. it also provides access to the *SYSTEM OPERATING PARAMETERS* and *SYSTEM DAQ CALIBRATION VALUES* panels. Figure 4 shows the *SYSTEM MANAGER CONTROL* panel.

The top blocks ("Chamber Pressure", "Pressure Cycles", "Chamber Temperature", and "Heating System") of the *SYSTEM MANAGER CONTROL* panel include digital indicators for chamber pressures, number of pressure cycles and temperatures. This block includes a message line ("System Messages") that is used to inform the operator of system progress and errors. There is also heater on/off control in the "Heating System" block; this control allows the operator to turn off the system heaters, thus preventing them from cycling around the temperature set point.

The "Independent Control" block consists of several buttons and slide switches that provide low-level control of system functions; many of the functions also have position indicators. These include:

Door Position Indicator: Provides current status ("Opened" or "Closed") of door position. Because the door is manually operated, no other controls/indicators are included.

Yoke Position Indicator: Provides current status ("Opened" or "Closed") of the yoke position. A single limit switch is used to determine yoke position; when the yoke is open, its position is absolutely known, but because the switch changes states well before the yoke is fully closed, its closed state is assumed.

Yoke "OPEN" Button: Allows the operator to open the yoke. Before this action is initiated, 120VAC power to the yoke-control circuitry is confirmed and pressure in the transfer chamber is checked; if there is no 120VAC power to the yoke circuitry or if the transfer-chamber pressure is above the maximum safe level, the system-manager request is ignored and the appropriate message is echoed to the message center

Yoke "CLOSE" Button: Allows the operator to close the yoke. Before this action is initiated, 120VAC power to the yoke-control circuitry and door position are confirmed; if there is no 120VAC power to the yoke circuitry or if the door is not properly closed, the system manager request is ignored and the appropriate message is echoed to the message center.

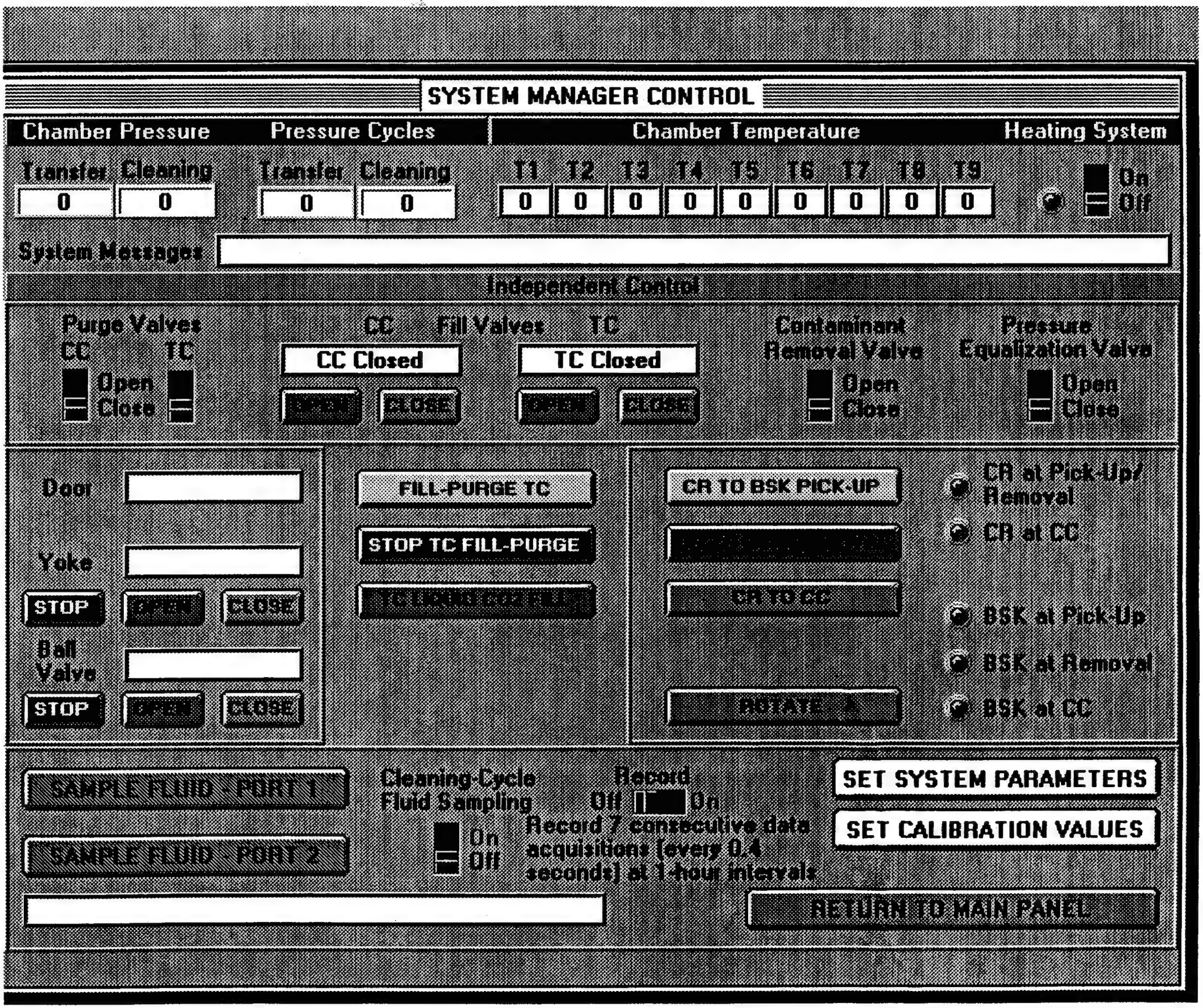


Figure 4: System Manager Control Panel

SYSTEM MANAGER CONTROL PANEL FUNCTIONS (CONT'D.)

Yoke "STOP" Button:	Allows the operator to immediately stop a yoke open or close sequence. No checking is performed before initiation of this action. Note that a manually operated emergency stop button is mounted on a bulkhead next to the yoke; this button can be pushed in to stop the yoke mechanism.
Ball-Valve Position Indicator:	Provides current status ("Opened", "Closed", "Opening", or "Closing") of the ball valve. States of relays in both the open and close circuit legs of the ball-valve actuator are monitored allowing determination of open/close/transition status.
Ball-Valve "OPEN" Button:	Allows the operator to open the ball valve. Before this action is initiated, 120VAC power to the ball-valve-actuator control circuitry is confirmed as is the differential pressure between the cleaning and transfer chambers; if there is no 120VAC power to the ball-valve-actuator circuitry or if the chamber-to-chamber differential pressure is too high, the system-manager request is ignored and the appropriate message is echoed to the message center
Ball-Valve "CLOSE" Button:	Allows the operator to close the ball valve. Before this action is initiated, 120VAC power to the ball-valve-actuator control circuitry is checked as is the position of the carriage. If there is no 120VAC power to the ball-valve-actuator circuitry or if the carriage is not in the cleaning chamber position, the system manager request is ignored and the appropriate message is echoed to the message center.
Ball-Valve "STOP" Button:	Allows the operator to immediately stop a ball-valve open or close sequence. No checking is performed before initiation of this action.
"CC" Purge Valve Slide Switch:	This switch allows the operator to open or close the two solenoid valves used to purge gas from the cleaning chamber. No checking of other system-manager activity or other open solenoid valves is performed before this action.

SYSTEM MANAGER CONTROL PANEL FUNCTIONS (CONT'D.)

"TC" Purge Valve Slide Switch: This switch allows the operator to open or close the solenoid valve used to purge gas from the transfer chamber. No checking of other system-manager activity or other open solenoid valves is performed before this action.

"CC Fill Valve" Position Indicator: This indicators shows the current position of the cleaning-chamber fill value ("CC Closed" or "CC Opened").

"CC OPEN" Button: This button allows the operator to open the two-way ball valve through which the cleaning chamber is filled with gas. Before opening the fill valve, the system checks the current positions of the ball valve and yoke; it also check the status of the 120 VAC power. If the both the ball valve and yoke are open or if a power failure is detected such that the valve cannot be operated or the positions of the yoke and ball valve cannot be confirmed, then the system ignores the request to open the fill valve. No checking of other system-manager activity or other open valves is performed before this action.

"CC CLOSE" Button: This button allows the operator to close the two-way ball valve through which the cleaning chamber is filled with gas. No checking of other system-manager activity or other open valves is performed before this action.

"TC Fill Valve" Position Indicator: This indicators shows the current position of the transfer-chamber fill value ("TC Closed" or "TC Opened").

"TC OPEN" Button: This button allows the operator to open the two-way ball valve through which the transfer chamber is filled with gas. Before opening the transfer fill valve, the system checks the status of the yoke and 120VAC power to the valve motor. If the yoke is opened or if a power failure is detected such that the valve cannot be operated or the position of the yoke cannot be confirmed, then the request to open the transfer-chamber fill valve is ignored. No checking of other system-manager activity or other open valves is performed before this action.

"TC CLOSE" Button: This button allows the operator to close the two-way ball valve through which the transfer chamber is filled with gas. No checking of other system-manager activity or other open valves is performed before this action.

SYSTEM MANAGER CONTROL PANEL FUNCTIONS (CONT'D.)

"Contaminant Removal Valve" Slide Switch: This switch allows the operator to open or close the solenoid valve used to remove particulate that collects in the bottom of the cleaning chamber. No checking of other system-manager activity or other open solenoid valves is performed before this action.

"Pressure Equalization Valve" Slide Switch: This switch allows the operator to open or close the solenoid valve used to equalize pressure between the cleaning and transfer chambers. No checking of other system-manager activity or other open solenoid valves is performed before this action.

"PURGE/FILL TC" Button: This button allows the operator to initiate the transfer-chamber purge/fill sequence. This sequence is identical to that used during an automatic cleaning cycle. Before staring the sequence, other system-manager activity and status of all solenoid and two-way valves is checked; if other system-manager activity is in progress or any valves are open the sequence is prevented from starting and a message is posted for the operator.

"STOP TC PURGE/FILL" Button: This button allows the operator to immediately stop a transfer-chamber fill-purge sequence. It is invisible until a transfer-chamber fill-purge cycle is started and remains visible until the operator terminates the procedure. No checking is before performing the action. Both the transfer-chamber purge and fill valves are closed.

"TC LIQUID CO_2 FILL" Button: This button allows the operator to start the liquid CO_2 fill portion of the fill-purge transfer-chamber procedure. The button is used so that the operator can record the weight of the liquid CO_2 cylinders before filling the transfer chamber. The button is invisible until completion of all fill-purge cycles associated with the fill-purge transfer-chamber procedure and remains visible only until the operator initiates the liquid CO_2 fill.

SYSTEM MANAGER CONTROL PANEL FUNCTIONS (CONT'D.)

Basket/Carriage Position LED Indicators: These five LED indicators show provide the operator with visible feedback about the location of the basket and carriage. LED indicators include "CR at Pick-Up/Removal", "CR at CC", "BSK st Removal", "BSK at Pick-Up", and "BSK at CC".

"CR TO BSK DROP" Button: This button allows the operator to initiate the sequence used to move the carriage to the basket release position. Before the sequence will start, voltage to the motors, voltage to limit switches, position of the ball valve, position of carriage, and status of other system-manager activity is checked; if no voltage is detected, or the ball valve is closed, or the carriage is already in the requested position, or another system-manager request is being performed, then the request is ignored and a message is posted.

"CR TO BSK PICKUP" Button: This button allows the operator to initiate the sequence used to move the carriage to the basket pickup position. Before the sequence will start, voltage to the motors, voltage to limit switches, position of the ball valve, position of carriage, and status of other system-manager activity is checked; if no voltage is detected, or the ball valve is closed, or the carriage is already in the requested position, or another system-manager request is being performed, then the request is ignored and a message is posted.

"CR TO CC" Button: This button allows the operator to initiate the sequence used to move the carriage to the cleaning-chamber position. Before the sequence will start, voltage to the motors, voltage to limit switches, position of carriage, and status of other system-manager activity is checked; if no voltage is detected, or the carriage is already in the requested position, or another system-manager request is being performed, then the request is ignored and a message is posted.

"ROTATE" Button: This button allows to operator to rotate the parts basket. The rotation scheme ("No Rotation", "90 Degree", or "Continuous Rotation") depends on the setting on the main panel.

SYSTEM MANAGER CONTROL PANEL FUNCTIONS (CONT'D.)

"Record" Button: This button turns on and off the data recording function. This recording function, unlike that on the main panel, immediately initiates recording activity. It is configured to record 75 data points at 0.4-second intervals (30 seconds of data) once every 60 minutes. Data is recorded to a default file - SCFDATA.DAT. Data recording continues until the switch is turned off, even if the operator returns to the main panel.

"SET SYSTEM PARAMETERS" Button: Allows the operator to access the panel containing important system operating parameters. A description of controls on this panel can be found in the SYSTEM OPERATING PARAMETER PANEL FUNCTIONS section of this manual.

"SET CALIBRATION VALUES" Button: Allows the operator to access the panel containing offset and sensitivity values for all system thermocouples and pressure transducers. A description of controls on this panel can be found in the SYSTEM DAQ CALIBRATION VALUES PANEL FUNCTIONS section of this manual.

"RETURN TO MAIN PANEL" Button: Allows the operator to return to the main panel after completion of tasks on the SYSTEM MANAGER CONTROL panel. Note that before the system will allow the operator to return to the main panel, all valves must be closed and all system-manager operations (i.e., yoke opening or closing, carriage in transit, etc.) must be completed.

Current Time/Date Indicator: Displays the current time and date for the operator. Note that there is no label on this indicator; it purpose is obvious when the program is running.

SYSTEM OPERATING PARAMETERS PANEL FUNCTIONS

The *SYSTEM OPERATING PARAMETERS* panel, shown in Figure 5, contains controls for important default operating parameters. Sections on this panel include "Transfer Chamber Purge/Fill Parameters", "Miscellaneous Parameters", "Heating Parameters", and "Fluid Sampling Parameters. The first section is labeled "Transfer Chamber Purge/Fill Parameters" and provides controls that can be used to set/reset critical parameters of a transfer-chamber fill/purge sequence. These parameters include the "Number of Purge Cycles" (number of purge/fill cycles to perform to insure minimal ambient atmosphere in the chamber), "Purge Intake Pressure (psi)" (the pressure at which the fill two-way ball valve is closed), and "Purge Exhaust Pressure (psi)" (the pressure at which the solenoid exhaust valve is closed). The next section contains miscellaneous controls including "Pressure Equalization Delta (psi)" (the pressure differential between the transfer and cleaning chambers at which the pressure-equalization solenoid valve is allowed to close), "Upper Temp Limit (deg C)" (the maximum temperature allowed before shut-down of system heaters), "High-Pressure Limit (psi)" (the pressure at which exhaust solenoid valves are opened to bleed off excessive chamber pressure). The "Heating Parameters" section contains temperature set-point controls for the heating system and include "Set Point (deg C)", "Upper Band Delta (deg C)" (defines the upper range of the proportional control band beyond which the heaters are off), "Lower Band Delta (deg C)" (defines the lower range of the proportional control band below which the heaters are on), and the "Heating Cycle Period (s)" (defines the length of a heater on off cycle within the proportional band; the on /off cycle lengths are determined from this interval and the current temperature). The last section contains the "Fluid Sampling Parameters" which are not yet included in the program. The "RETURN TO SYSTEM MANAGER" button allows the user to return to the panel specified on the button. A current time/date indicator is included at the bottom of the panel.

SYSTEM DAQ CALIBRATION VALUES PANEL FUNCTIONS

The *SYSTEM DAQ CALIBRATION VALUES* panel (refer to Figure 6) contains controls for setting/changing the sensitivities and offsets of the system thermocouples and pressure transducers. There are eleven sensitivity and eleven offset controls, one for each of the nine thermocouples and two pressure transducers. There use is self-explanatory; no changes should be made only after system calibration and only if absolutely necessary. The "RETURN TO SYSTEM MANAGER" button allows the user to return to the panel specified on the button. A current time/date indicator is included at the bottom of the panel.

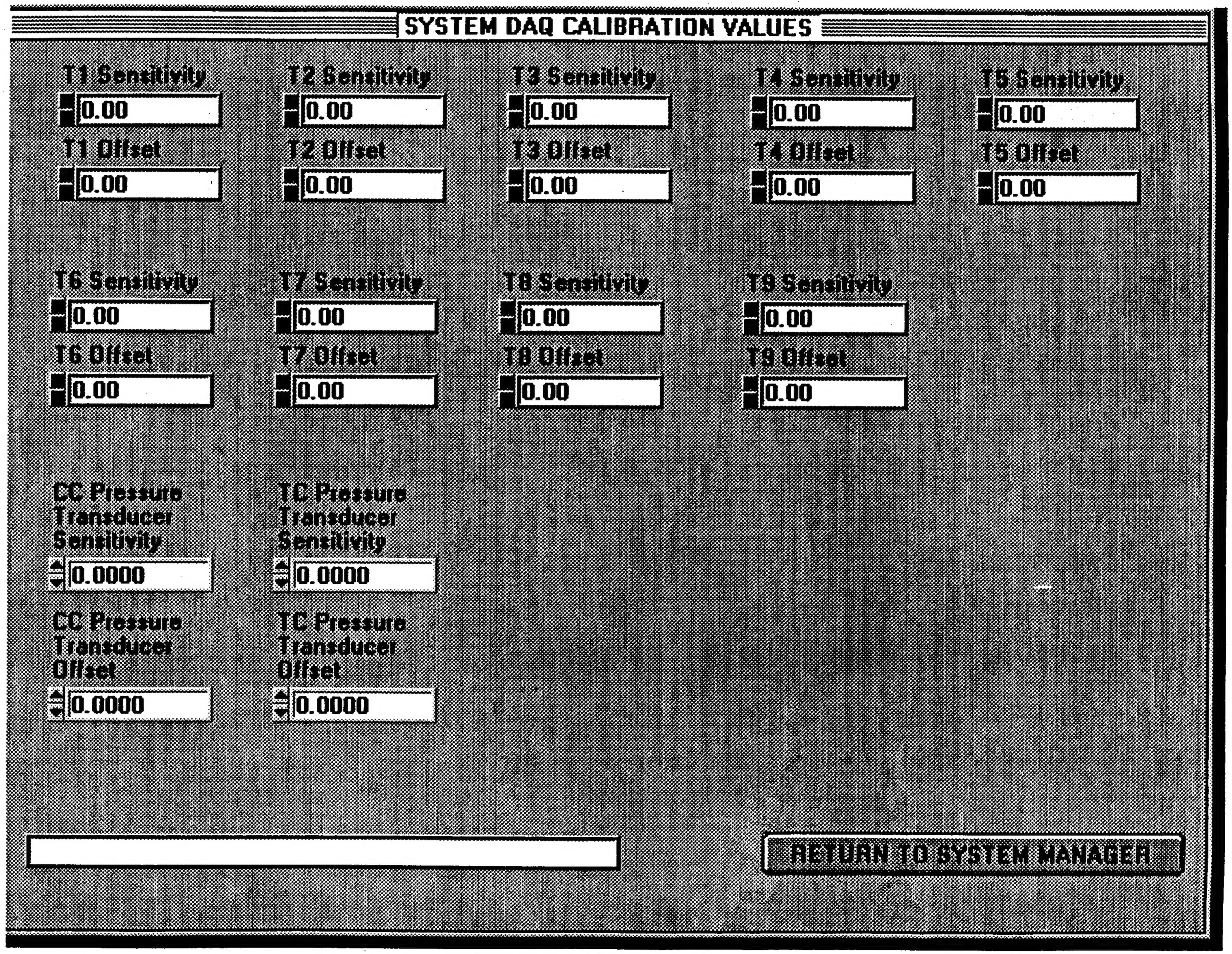


Figure 6. System DAQ Calibration Values Panel